UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

WASHINGTON, D.C. 20460

OFFICE OF
CHEMICAL SAFETY AND
POLLUTION PREVENTION

MEMORANDUM

Date: March 29, 2016

SUBJECT: **Dicamba.** Bridging Data Demonstrating DGA (diglycolamine), BAPMA (N,N-Bis-(3-aminopropyl) methylamine) and DETA (diethylenetriamine) Salt Product Equivalency, and the Independent Laboratory Validation of the BASF Method Developed for Determining Dicamba Residues in Crops. Abbreviated Residue Chemistry Review.

PC Code: 100094	**DP Barcode:** D429868 & D429964
Decision No.: 463710	**Registration No.:** 524-582 & 7969-GUL
Petition No.: NA	**Regulatory Action:** R170 New Food-Use Registrations
Risk Assessment Type: NA	**Case No.:** 0065
TXR No.: NA	**CAS No.:** 1918-00-9
MRID Nos.: 49379301-49379305	**40 CFR:** §180.227

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Executive Summary

The BASF Corporation has submitted bridging data to support the requested new use registration of its Engenia herbicide, a N,N-Bis-(3-aminopropyl) methylamine (BAPMA) salt formulation of dicamba. The proposed Engenia label (EPA Reg. No. 7969-GUL) has been revised to reflect current application requirements and to include its use on dicamba/glufosinate-tolerant cotton and dicamba-tolerant soybean. Dicamba is a selective benzoic acid herbicide used for controlling weeds prior to their

emergence. It is available for use in either acid or salt forms with registered uses being maintained on a wide variety of crop and livestock commodities. In support of this request, bridging data for pasture grasses, wheat, field corn, and soybeans were provided to demonstrate equivalency of residues on conventional crops resulting from the BAPMA salt product with respect to the registered diglycolamine (DGA) salt. Included in the bridging data are results for another new formulation of dicamba containing diethylenetriamine (DETA) salt which are also presented in this summary. Registration of the DETA formulation of dicamba is not being requested at this time and no proposed product label has been provided with these data for evaluation. For the use of the BAPMA product on dicamba/glufosinate-tolerant cotton and dicamba-tolerant soybean, BASF has obtained a limited right to cite the relevant Monsanto data developed for their diglycolamine salt M1691 Herbicide formulation (EPA Reg. No. 524-582) for this application. An independent laboratory validation of the liquid chromatography/mass spectrometer/mass spectrometer (LC/MS/MS) method, BASF Method D0902, used for analyzing the field trial samples in the bridging studies is also included for assessment. This memo summarizes the regulatory conclusions following review of these data.

Regulatory Recommendations

To support registration of the new BAPMA salt formulation of dicamba, side-by-side field trials on representative crops were made to show product equivalency. Bridging studies provided for pasture grasses, wheat, field corn, and soybeans show that the average combined residues of dicamba are similar in the various end-use products tested with all results falling well below established tolerance limits. Provided the label is amended as specified below, there are no residue chemistry considerations that would preclude registration of the new BAPMA salt formulation of dicamba. Included in the bridging data are results for another new dicamba end-use product formulated as a DETA salt soluble liquid concentrate (SL). Comparable residues were obtained for the DETA formulation but use of this product has not been requested. Subsequently, when registration of the DETA product is sought, these bridging data are sufficient to support its use but a proposed label must be provided to the Agency for review. In regard to the assessment of BASF Analytical Method D0902, HED determines it to be adequate for the tolerance enforcement of crops pending that the editorial revisions recommended by the independent validation laboratory are addressed.

A human-health risk assessment is forthcoming.

<u>860.1200 Directions for Use</u>

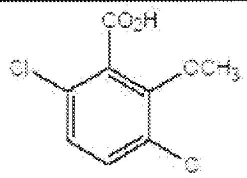
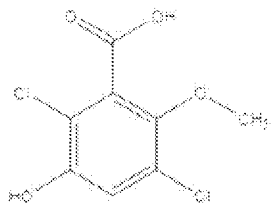
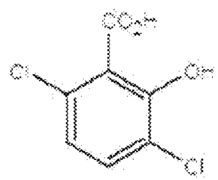
An amended Section B is required noting that no more than two (2) post-emergence applications may be made past the first open boll stage when treating dicamba-tolerant cotton is allowed.

Background

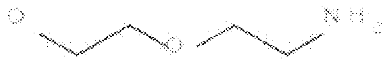
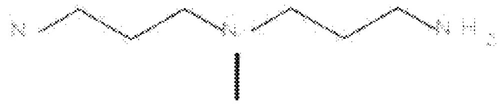
<u>Chemical Identity</u>

The chemical identity of the dicamba residues of concern which include the dicamba parent compound and the 5-hydroxy dicamba and 3,6-dichlorosalicylic acid metabolites are presented below in Table 1.

Table 1. Test Compound Nomenclature: Dicamba and its Residues of Concern.

Compound	
Common name	Dicamba
Company experimental name	MON 11900
IUPAC name	3,6-dichloro-*o*-anisic acid or 3,6-dichloro-2-methoxybenzoic acid
CAS name	3,6-dichloro-2-methoxybenzoic acid
CAS registry number	1918-00-9 (dicamba acid), 104040-79-1 (diglycolamine salt), or 53404-28-7 (monoethanolamine salt)
End-use product	Clarity® Herbicide: SL formulation containing 4 lb ae/gal
Compound	
Common name	5-Hydroxy-dicamba
Company experimental name	5-OH dicamba
IUPAC/CAS name	2,5-dichloro-3-hydroxy-6-methoxybenzoic acid
CAS registry number	7600-50-2
Compound	
Common name	DCSA: 3,6-dichlorosalicylic acid
Company experimental name	MON 52708
IUPAC/CAS name	3,6-dichloro-2-hydroxybenzoic acid
CAS registry number	3401-80-7

The DGA and BAPMA salts are counter ions used in the formulation of popular herbicide end-use products such as dicamba. The physiochemical properties of the DGA and BAPMA salts are presented in Table 2.

Table 2. Physiochemical Properties of the DGA and BAPMA Salts.[1]		
Phys./Chem Properties	**DGA**	**BAPMA**
Structure		
Chemical Name	diethylene glycol monoamine	N,N-Bis-(3-aminopropyl) methylamine
CAS No.:	929-06-6	105-83-9
Molecular Wt. (g/mol)	105.14	145.25
Log Kow	-1.8866	-0.9390
BP (°C)	224	232.5
MP (°C)	13.94	45.26
VP (mm Hg)	0.0246 (Antoine)	0.0425 (Grain)
Water Sol. (mg/L)	1E+006	1E+006
Henry's Law Conc. 25°C	2.13E-013 atm	1.54E-013 atm
Log Koa	7.74	9.4
Air Half-Life (Hrs)	1.8	0.83
Koc (L/kg)	1	110
Log BCF	0.5	0.5

[1] BASF Comparison of Physical/Chemical Properties for BAPMA and DGA Salts Using The Estimations Programs Interface for Windows (EPI Suite) QSAR Tool (07/21/2015).

Detailed Discussion

Pesticide Use Pattern/Directions for Use

The Engenia Herbicide (EPA Reg. No. 7969-GUL) is a new BAPMA salt formulation of the dicamba herbicide developed by the BASF Corporation. It is an SL product proposed for use in treating conventional crops, as well as dicamba/glufosinate-tolerant cotton and dicamba-tolerant soybean. This end–use product contains 48.38% active ingredient in the form of the BAPMA salt of dicamba (equivalent to 5.0 lb ae/gal). The proposed BAPMA label depicts the same pattern of use found on the labels of the many other registered salts of dicamba which allows growers great flexibility for application to control weeds when cultivating crops. Table 3 provides a summary of the use directions taken directly from the proposed BAPMA product label.

Table 3. Summary of Directions for Use of the Engenia 5.0 lb ae/gal SL Herbicide Formulation of Dicamba (EPA Reg. No. 7969-GUL).							
Application. Timing, Type, and Equip.	**Max. Applic. Rate (lb ae[1]/A)**	**Max. No. Applic. per Season**	**Max. Seasonal Applic. Rate (lb ae/A)**	**Combined Max. Seasonal Applic. Rate (lb ae/A)**	**RTI[2] (days)**	**PHI[3] (days)**	**Use Directions and Limitations[4]**
Asparagus							
Post-emergence Broadcast (40-60 gal/A)	0.5	NS[5]	0.5	NA[6]	1	1	- Apply in combination with glyphosate (Roundup). - Do not harvest 24 hours after treatment. - Do not use in the Coachella Valley of CA.
Post-harvest, Broadcast (Burndown Treatment)	1.0		2.0				

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Corn (field, seed, silage) and Popcorn							
Preplant-Pre-emergence Broadcast Pre-emergence Broadcast Post-Emergence Broadcast	0.5	NS	0.5	1.5	14	NS	- Engenia is not registered on sweet corn. - Do not contact seeds. - Application rates vary by soil texture and growth stage. - Adjuvants may be used and it can be mixed with other herbicide products.
Cotton (non-dicamba-tolerant)							
Pre-emergence Broadcast (Preplant Burndown)	0.25	NS	0.25	0.25	NA	NA	- Adjuvants may be used and it can be mixed with other herbicide products. - After application, wait for 1" of rainfall/irrigation and a 21-day interval before planting. - For MO and TN only, wait for 1" of rainfall/irrigation and a 14-day interval before planting.
Cotton (dicamba/glufosinate-tolerant)							
Pre-emergence Broadcast	1.0	NS	1.0	2.0	14	7	- Use of a COC or MSO adjuvants are only used when other products require them. For best results apply at min spray rate of 10 GPA.
Post-emergence, Broadcast	0.5	NS	2.0				
Grass Grown for Seed							
Post-Emergence Broadcast	1.0	NS	2.0	NA	NS	37-51	- Apply with recommended adjuvants to seedling grasses when the crop reaches the 3-5 leaf stage. - Follow listed grazing restrictions; 7 days for 0.5 lb ae/A applications and 21 days for 1.0 lb ae/A applications. - Follow listed PHI restrictions; 37 days for 0.5 lb ae/A applications and 51 days for 1.0 lb ae/A applications.

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Application, Timing, Type, and Equip.	Max. Applic. Rate (lb ae[1]/A)	Max. No. Applic. per Season	Max. Seasonal Applic. Rate (lb ae/A)	Combined Max. Seasonal Applic. Rate (lb ae/A)	RTI[2] (days)	PHI[3] (days)	Use Directions and Limitations[4]
Pasture, Hay, Rangeland, and Farmstead (non-cropland)							
Broadcast treatment of non-cropland	0.5	NS	1.0	NA	NA	NA	- Engenia can be applied using water, oil-in-water emulsions including invert systems, or sprayable fluid fertilizer as a carrier. - Follow listed grazing restrictions: 7 days for 0.5 lb ae/A applications and 21 days for 1.0 lb ae/A applications.
Broadcast treatment of small grain grown for pasture & newly seeded grass	0.5		0.5				
Spot Treatment	0.001 -5ft canopy 0.04 – 10 ft canopy 0.09 – 15ft canopy		NS				- Apply as a cut surface treatment for unwanted trees and prevention of sprouts of cut trees. - Apply as an undiluted spot treatment directly to the soil or as a Lo-Oil basal bark treatment using an oil-in-water emulsion solution when plants are dormant.
Proso Millet							
Broadcast and spot treatment for broadleaf weed control	0.125	NS	0.125	NA	NS	NS	- Apply to actively growing weeds when proso millet is in the -2 to 5-leaf stage. - Apply only if proso millet injury is acceptable. - Follow listed grazing restrictions: 7 days for 0.5 lb ae/A.
Small Grain (Barley, Oats, Triticale & Wheat)							
Post-emergence, Broadcast - Barley	0.094 – up to 4-leaf stage	NS	0.34 spring seeded barley 0.38 fall seeded barley	NA	NS	7 and 37-hay	- May be applied before, during or after planting. - Do not apply pre-harvest in CA. - Do not use a COC for post-emergence application. - Application in periods of rapid growth may result in temporary crop leaning. - Do not apply if there potential for crop injury is unacceptable. - Do not graze small grain within 7 days of treatment.
Post-emergence, Broadcast - Oats	0.125 – up to 5-leaf stage		0.125				
Post-emergence, Broadcast – Wheat & Triticale	0.125 – up to the 6-leaf stage		0.5				
Post-emergence, Broadcast – Fall Seeded Wheat	0.25 – after the 3-leaf stage						

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Application. Timing, Type, and Equip.	Max. Applic. Rate (lb ae[1]/A)	Max. No. Applic. per Season	Max. Seasonal Applic. Rate (lb ae/A)	Combined Max. Seasonal Applic. Rate (lb ae/A)	RTI[2] (days)	PHI[3] (days)	Use Directions and Limitations[4]
Sorghum							
Pre-emergence Broadcast	0.25	NS	0.5	0.5	NS	30	- May be used pre-plant, post-emergence or pre-harvest in split applications. - Pre-plant treatments are to be made 14-days before planting. - Application in periods of rapid growth may result in temporary crop leaning. - Do not graze before mature grain stage or within 7-days of treatment.
Post-emergence, Broadcast							
Soybean (non-dicamba-tolerant)							
Pre-plant Broadcast Spray	0.25 @ a 14-day pre-plant Interval 0.5 @ a 28-day pre-plant Interval	NS	0.5	2.0	NS	7	- Application can be made with other herbicides. - Pre-plant application cannot exceed 0.5 lbs ae/A per season. - Do not apply in areas where annual rainfall is below 25 inches. - Pre-harvest applications are only made when pods are a mature brown color. - Do not make pre-harvest applications in CA. - Do not feed pre-harvest treated fodder and hay.
Pre-harvest Broadcast Spray	1.0		2.0				
Soybean (dicamba-tolerant)							
Pre-emergence Broadcast	1.0	NS	1.0	2.0	NS	7-forage And 14-hay	- Application can be made with other herbicides. - Pre-emergent applications can be made at 1.0 lbs ae/A to medium to fine texture soils and at 0.5 lbs ae/A to course and sandy soils. - Do not apply after first bloom. - Post-emergent treatments may cause wilting for 24-72 hours afterwards. - Do not apply post-emergent treatments by aerial application. - Do not apply with ammonium containing additives.
Post-emergence, Broadcast	0.5	2	1.0				

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Sugarcane							
Post-emergence, Broadcast	1.0	NS	2.0	NA	NS	87	- Application can be made with other herbicides. - Application may be made any time after weed emergence. - When possible spray beneath the sugarcane canopy to minimize the likelihood of crop injury.
Farmstead Turf (non-cropland) and Sod Farms							
Broadcast treatment	0.5	NS	1.0	NA	NS	NS	- To avoid injury to newly seeded grasses, delay application until after the second mowing. - Application may cause stunting and discoloration in sensitive grasses (bentgrass, buffalograss, carpetgrass & St. Augustinegrass). - Do not use on residential sites. - Applications can be made at 0.25 lbs ae/A to medium to fine texture soils and at 0.125 lbs ae/A to course and sandy soils.

[1] ae = Acid Equivalents
[1] RTI = Re-Treatment Interval
[2] PHI = Pre-Harvest Interval
[3] COC = Crop Oil Concentrate; MSO = Methylated Seed Oil.
[4] NS = Not Specified
[5] NA = Not Applicable

Conclusions. The proposed use directions for the new BAPMA salt formulation of dicamba are adequate to allow evaluation of the submitted residue data. The residue data provided examine the broad use pattern of dicamba using post-emergence broadcast treatments made at the maximum application rate following the specified minimum pre-harvest interval (PHI). Prior residue data submitted for dicamba-tolerant cotton shows that later post-emergence treatments give much higher residues then those made at earlier growth stages. Following the pattern of late season use demonstrated by the field trial data, no more than two (2) post-emergence applications may be made after the first open boll stage when treating dicamba-tolerant cotton. Therefore, the BAPMA product label must be amended to include this restriction since the data provided only support this pattern of use. A proposed label was not provided in support of the data included for the new DETA salt formulation of dicamba. Although registration of the DETA product is not being sought, a proposed label must be provided to the Agency for review when its registration is subsequently requested.

Methods

DER Reference: 49379301.der.doc (Analytical Methods for Plant Matrices)

The BASF Corporation has submitted an independent laboratory validation (ILV) of the LC/MS/MS method used for analyzing the field trial samples in the bridging studies. This method is titled, BASF Analytical Method D0902: The Determination of Residues of Dicamba (BAS 183 H) and its Metabolite, 5-Hydroxy-Dicamba in Corn Matrices Using LC/MS/MS. It was previously reviewed as a data collection method used to acquire crop field trial data for corn (D375578, A. Kamel, 04/30/2013). The validated limit of quantitation (LOQ) determined as the lowest level of method validation (LLMV) is reported to be 0.01 ppm for both dicamba analytes in all corn matrices. The current ILV study was performed by ADPEN Laboratories, Inc. using samples of untreated corn grain and stover fortified with dicamba and 5-OH dicamba each at 0.01 and 1.0 ppm (LOQ and 100x LOQ).

For this method, homogenized crop samples are heated with 1 N HCl at ~90 °C for ~45 minutes. The extract is cooled to room temperature and filtered, then adjusted to volume with water. An aliquot of the extract is adjusted to pH 9-10 with concentrated NH_4OH, vortexed, and adjusted to pH 3-4 with concentrated formic acid. Sodium chloride is added, and the extract is partitioned twice with hexane:ethyl acetate (1:1, v:v) followed by centrifugation. The resulting organic phase is reduced to dryness under nitrogen, then reconstituted in methanol:water (10:90, v:v) for analysis by LC/MS/MS. The precursor/product ion transitions monitored for dicamba originated from the same ion fragment at *m/z* 218.9 and were 218.9 →174.8 and 220.9→176.8 (the isotope of *m/z* 218.9). However, three different precursor/product ion transitions were monitored for 5-OH dicamba at *m/z* 234.9→154.9, 234.9→190.7, and 236.9→192.7. The first set of transitions is monitored for quantitation purposes, and the second and third set of transitions are monitored for confirmatory purposes. The confirmatory ion transition may be used for quantitation if interferences are found with the quantitation ion transition. The method does not provide for conversion of residues of 5-OH dicamba to parent equivalents. Quantifiable residues may be converted to parent equivalents using molecular weight conversion factor of 0.9325 (MW dicamba 221.04/MW 5-OH dicamba 237.04).

In all, the ILV resulted in acceptable mean recoveries ranging from 77.9-95.1% obtained for the analysis of corn grain and stover after the first trial. Contact with the sponsor was not required. The validating laboratory did not identify any critical steps except to make several editorial recommendations and suggestions. The ILV laboratory reported that the method procedure could be completed in 8 hours (1 calendar day) for a set of 12 samples each, without counting the overnight LC/MS/MS analysis time. The method is suitable for additional number of samples, which may be taken through the procedure at one time.

Conclusions: The LC/MS/MS method used for analyzing the field trial samples in the bridging studies has undergone successful ILV study. In prior proficiency testing, acceptable method validation data were obtained in corn matrices fortified with a mixed standard of dicamba and 5-OH dicamba adequate in bracketing expected residues (D375578, A. Kamel, 04/30/2013). Because the method uses acid hydrolysis procedures similar to those used in the established enforcement methods, radiovalidation data are not required for verification of extraction efficiency (48001303.der.doc). Following the criteria specified in the Tolerance Method Validation checklist (SOP No. ACB 019, Revision 1.0), BASF Analytical Method D0902 meets these conditions depicting the suitability of an enforcement methodology. It is detailed in its approach using an established instrumental technique and common laboratory procedures to perform sample analysis. Test results are considered to be sufficient as illustrated by the supporting chromatograms and calculations provided in the supporting data submissions. Pending editorial revisions recommended by the independent validation laboratory, HED determines the BASF method to be adequate for the tolerance enforcement of crops.

Bridging Data

The BASF Corporation has submitted field trial data for dicamba on pasture grass, wheat, field corn, and soybean. These studies were made to demonstrate the equivalency of the new 4 lb ae/gal (480 g ae/L) DETA salt (BAS 183 UYH) and 5 lb ae/gal (600 g ae/L) BAPMA salt (BAS 183 WBH) SL formulations of dicamba. Four (4) field trials were conducted with each crop in the United States during the 2010 growing season. They were made as side-by-side bridging trials along with the Clarity Herbicide (EPA Reg. No. 7969-1374) which is a registered 4 lb ae/gal (480 g ae/L) diglycolamine (DGA) salt (BAS 183-09) SL formulation of dicamba. Each trial site consisted of one untreated plot (Plot 1) and three side-by-side treated plots (Plots 2, 3, and 4). For study, the three treated test plots generally received post-emergence broadcast treatment made at the maximum application rate using one of the three SL formulations of dicamba. The minimum PHI was followed for harvesting. The residues of concern for corn, grasses and wheat, i.e. dicamba and its 3,6-dichloro-5-hydroxybenzoic acid (5-OH dicamba) metabolite, were measured. The residues of concern in/on soybeans, i.e. dicamba, 5-OH dicamba and 3,6-dichlorosalicylic acid (DCSA) were measured as well. Total residues expressed as parent equivalents are reported below.

Pasture Grasses
DER Reference: 49379302.der.doc (Magnitude of the Residue in Pasture Grasses; Formulation Bridging Study)

The four (4) field trials for pasture grass were conducted in North American Free Trade Agreement (NAFTA) Growing Zones 3 (FL; 1 trial on Bahiagrass), 5 (MO and NE; 1 trial each on Tall Fescue and Smooth Brome, respectively), and 10 (CA; 1 trial on Bermudagrass). Each treated plot received one foliar broadcast application at 0.983-1.02 lb acid equivalent (ae)/A (1.10-1.15 kg ae/ha) using one of the three SL formulations of dicamba. Applications were made using ground equipment, in spray volumes of ~20 gal/A (185-193 L/ha) of water. A nonionic surfactant (NIS) was added to spray mixtures for each trial. Samples of grass forage and hay were harvested at PHIs of 0, 7, 14, 21, 28/29, and 56 days. Samples of hay were allowed to dry in the field for 2-7 days. In the FL trial, samples of forage from Plot 2 were collected 0-3 days after cutting.

The field trial samples were stored frozen a maximum of 328 days (10.8 months) for forage and 337 days (11.1 months) for hay from harvest to extraction. Samples were analyzed for the dicamba parent and 5-OH dicamba residues of concern within 2 days of extraction. Adequate storage stability data are available to show that residues of dicamba and 5-OH dicamba are stable during frozen storage in/on grass forage and hay for up to 314 and 320 days to support this study (D317699, C. Olinger, 12/20/2005).

The grass samples were analyzed for residues of dicamba and its 5-OH dicamba metabolite using LC/MS/MS method PSL RA006 which is based upon BASF Method D0902 with minor modifications. The LOQ of the method determined as the LLMV is reported to be 0.01 ppm for all the dicamba analytes in each grass matrix. The method was verified prior to and in conjunction with the grass sample analysis to demonstrate it was adequate for data collection. Fortification levels for the concurrent method recovery analyses were adequate in bracketing the expected residue levels within an order of magnitude. Quantifiable residues of 5-OH dicamba were converted to parent equivalents by the registrant using molecular weight conversion factor of 0.9325.

Following the side-by-side testing performed to compare the DETA and BAPMA end-use products to the registered DGA formulation, residue levels were found to be similar in the raw agricultural commodities (RACs) of pasture grass (Table 4). At the 21-day PHI following the labeled grazing and haying restrictions required for applications of up to 1.0 lb ae/A, the average combined residues in grass forage and hay were found to be comparable for each product. In grass forage, the average combined residues were 26.12 ppm for the DGA salt, 19.84 for the DETA salt, and 21.81 ppm for the BAPMA salt. The average combined residues in grass hay were 35.11 ppm for the DGA salt, 28.26 ppm for the DETA salt, and 32.20 ppm for the BAPMA salt. In the residue decline trials, combined residues of dicamba and the 5-OH dicamba metabolite were observed to decrease with increasing PHIs in/on forage and hay.

Table 4. Summary of Residues from Grass Field Trials with Dicamba.

Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
				Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Grass Forage										
4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	21	4	12.33	43.31	12.59	43.00	24.52	26.12	15.58
4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	7.50	33.36	7.63	32.25	19.84	19.89	14.00
5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	9.17	34.71	9.44	33.50	22.15	21.81	11.59
Grass Hay										
4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	21	4	19.94	55.02	24.52	52.85	35.11	36.90	12.37
4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	19.32	50.90	20.00	47.11	28.26	30.91	12.09
5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	19.49	53.27	20.26	47.53	32.20	33.05	11.21

[1] n = number of field trials.
[2] Values based on total number of samples.
[3] Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation. For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (0.01 ppm).

Wheat

DER Reference: 49379303.der.doc (Magnitude of the Residue in Wheat; Formulation Bridging Study)

The four (4) field trials for wheat were conducted in NAFTA Growing Zones 5 (IA and MN, 2 trials), 7 (NE, 1 trial), and 11 (WA, 1 trial). Each treated plot received one preplant soil application at 0.123-0.132 lb acid equivalent (ae)/A (0.138-0.148 kg ae/ha) followed by two foliar broadcast applications at 0.241-0.262 lb ae/A/application (0.270-0.293 kg ae/ha/application). The second application was made at the six-leaf growth stage, 31-45 days after the preplant application. There was a 50- to 68-day retreatment interval (RTI) between the second and third foliar applications. Applications were made using ground equipment, in spray volumes of ~14-20 gal/A (128-188 L/ha) of water. A NIS was added to spray mixtures for each trial. The total application rates achieved for study were 0.366-0.388 lb ae/A (0.410-0.434 kg ai/ha) for wheat forage and hay and 0.615-0.650 lb ae/A (0.689-0.724 kg ae/ha) for grain and straw. Samples of wheat forage and hay were harvested at a PHI of 7 days after the second application. Hay was allowed to dry in the field for 5-8 days after harvest. Samples of wheat grain and straw were harvested at a PHI of 6-7 days after the third application.

The field residue samples were stored frozen a maximum of 310 days (10.2 months) for forage, 307 days (10.1 months) for hay, 996 days (32.8 months) for grain, and 960 days (31.6 months) for straw from harvest to extraction. Samples were analyzed for the dicamba parent and 5-OH dicamba residues of concern within 0-16 days of extraction. Adequate storage stability data are available to show that residues of dicamba and 5-OH dicamba are stable during frozen storage in/on wheat forage and hay for up to 258 and 283 days (8.5 and 9.3 months), respectively, as well as in field corn forage, silage, grain, and fodder for up to 2 years, respectively (D317699, C. Olinger, 12/20/2005).

The wheat samples were analyzed for residues of dicamba and its 5-OH dicamba metabolite by LC/MS/MS analysis using BASF Method D0902 (and BASF REG DOC No. 97/5441) with minor modifications. The LOQ of the method determined as the LLMV is reported to be 0.01 ppm for all the dicamba analytes in each wheat matrix. The method was verified prior to and in conjunction with the grass sample analysis to demonstrate it was adequate for data collection. Fortification levels for the concurrent method recovery analyses were adequate in bracketing the expected residue levels within an order of magnitude. Quantifiable residues of 5-OH dicamba were converted to parent equivalents by the registrant using molecular weight conversion factor of 0.9325.

Following the side-by-side testing performed to compare the DETA and BAPMA end-use products to the registered DGA formulation, residue levels were found to be similar in wheat RACs (Table 5). At the 7-day PHI following the labeled grazing and haying restrictions required for applications of up to 0.5 lb ae/A, the average combined residues in wheat forage and hay were comparable for each product. In wheat forage, the average combined residues were 5.10 ppm for the DGA salt, 4.77 ppm for the DETA salt, and 4.45 ppm for the BAPMA salt. The average combined residues in wheat hay were 12.8 ppm for the DGA salt, 11.2 ppm for the DETA salt, and 10.5 ppm for the BAPMA salt. At the 7-day PHI required for the harvesting of grain, combined residues were 0.668 ppm for the DGA salt, 0.359 ppm for the DETA salt, and 0.334 ppm for the BAPMA salt. In wheat straw at the 7-day PHI, combined residues were 15.2 ppm for the DGA salt, 10.1 ppm for the DETA salt, and 8.4 ppm for the BAPMA salt. No residue decline trials were conducted for wheat.

Table 5. Summary of Residues from Wheat Field Trials with Dicamba.

Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
				Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Wheat Forage										
4 lb ae/gal DGA SL	0.366-0.387 [0.410-0.433]	7	4	3.89	7.00	3.96	6.57	4.94	5.10	1.26
4 lb ae/gal DETA SL	0.372-0.388 [0.417-0.434]	7	4	3.71	5.61	4.32	5.41	4.69	4.77	0.490
5 lb ae/gal BAPMA SL	0.372-0.378 [0.417-0.423]	7	4	3.10	5.73	3.15	5.28	4.55	4.45	0.941
Wheat Hay										
4 lb ae/gal DGA SL	0.366-0.387 [0.410-0.433]	7	4	8.15	17.2	8.42	16.8	13.0	12.8	3.41
4 lb ae/gal DETA SL	0.372-0.388 [0.417-0.434]	7	4	6.13	14.5	6.46	13.3	12.6	11.2	3.21
5 lb ae/gal BAPMA SL	0.372-0.378 [0.417-0.423]	7	4	6.23	14.2	6.56	13.7	10.8	10.5	3.05
Wheat Grain										
4 lb ae/gal DGA SL	0.616-0.649 [0.690-0.726]	6-7	4	<0.0919	1.76	0.0160	1.73	0.393	0.668	0.737
4 lb ae/gal DETA SL	0.624-0.650 [0.699-0.727]	6-7	4	<0.0619	0.911	<0.0890	0.863	0.243	0.359	0.346
5 lb ae/gal BAPMA SL	0.615-0.630 [0.689-0.705]	6-7	4	<0.0827	0.929	<0.0979	0.905	0.167	0.334	0.384
Wheat Straw										

Table 5. Summary of Residues from Wheat Field Trials with Dicamba.										
Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
				Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
4 lb ae/gal DGA SL	0.616-0.649 [0.690-0.726]	6-7	4	6.72	32.0	7.65	31.7	10.8	15.2	11.1
4 lb ae/gal DETA SL	0.624-0.650 [0.699-0.727]	6-7	4	5.72	27.9	6.04	27.7	9.05	13.0	10.1
5 lb ae/gal BAPMA SL	0.615-0.630 [0.689-0.705]	6-7	4	3.71	24.6	4.85	24.3	10.2	12.4	8.4

[1] n = number of field trials.
[2] Values based on total number of samples.
[3] Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation. For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (0.01 ppm).

Field Corn

DER Reference: 49379304.der.doc (Magnitude of the Residue in Field Corn; Formulation Bridging Study)

The four (4) field trials for field corn were conducted in NAFTA Growing Zone 5 (IA, and MN; 1 trial each, and NE; 2 trials). Each treated plot received one preplant soil application at 0.489-0.515 lb acid equivalent (ae)/A (0.548-0.577 kg ae/ha) followed by two foliar broadcast applications made at 0.482-0.508 lb ae/A (0.540-0.569 kg ae/ha) and 0.242-0.262 lb ae/A (0.271-0.293 kg ae/ha), respectively. The second application was made at the V3 (BBCH 13) growth stage 17-23 days after the preplant application. The third application was made at the V7-V9 (BBCH 18) growth stage with an 18- to 34-day RTI between the second and third foliar applications. Applications were made using ground equipment, in spray volumes of ~15-20 gal/A (139-190 L/ha) of water. A NIS was added to spray mixtures for each trial. The total application rates achieved for study was 1.22-1.28 lb ae/A (1.37-1.43 kg ae/ha). Samples of corn forage were harvested at a PHI of 43-52 days and samples of corn grain and stover were harvested at a PHI of 90-99 days after the last foliar application.

The field residue samples were stored frozen a maximum of 574 days (18.9 months) for forage, 538 days (17.7 months) for grain, and 541 days (17.8 months) for stover from harvest to extraction. Samples were analyzed for the dicamba parent and 5-OH dicamba residues of concern on the same day of extraction. Adequate storage stability data are available to show that residues of dicamba and 5-OH dicamba are stable during frozen storage for up to 2 years in/on field corn forage, silage, grain, and fodder (D317699, C. Olinger, 12/20/2005).

The field corn samples were analyzed for residues of dicamba and its 5-OH dicamba metabolite using LC/MS/MS method PSL RA006 which is based upon BASF Method D0902 with minor modifications. The LOQ of the method determined as the LLMV is reported to be 0.01 ppm for all the dicamba analytes in each corn matrix. The method was verified prior to and in conjunction with the grass sample analysis to demonstrate it was adequate for data collection. Fortification levels for the concurrent method recovery analyses were adequate in bracketing the expected residue levels within an order of magnitude. Quantifiable residues of 5-OH dicamba were converted to parent equivalents by the registrant using molecular weight conversion factor of 0.9325.

Following the side-by-side testing performed to compare the DETA and BAPMA end-use products to the registered DGA formulation, residue levels were found to be similar in corn RACs (Table 6). At a PHI of 43-52 days for the harvesting of corn forage, average combined residues in this RAC were 0.371 ppm for the DGA salt, 0.361 for the DETA salt, and 0.415 for the BAPMA salt. At the PHI of 90-99 days, average combined residues in corn grain were 0.0207 ppm for the DGA salt, 0.02 ppm for the DETA salt, and 0.02 ppm for the BAPMA salt. In corn stover, average combined residues were 0.208 for the DGA salt, 0.238 ppm for the DETA salt, and 0.266 for the BAPMA salt. No residue decline trials were conducted for field corn.

Table 6. Summary of Residues from Corn Field Trials with Dicamba.

Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
				Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Corn Forage										
4 lb ae/gal DGA SL	1.24-1.28 [1.39-1.43]	43-52	4	<0.02	0.667	<0.239	<0.504	0.370	0.371	0.150
4 lb ae/gal DETA SL	1.22-1.28 [1.37-1.43]	43-52	4	<0.0711	0.777	<0.0836	0.707	0.327	0.361	0.261
5 lb ae/gal BAPMA SL	1.23-1.26 [1.37-1.42]	43-52	4	<0.0471	0.762	<0.0594	0.623	0.489	0.415	0.268
Corn Grain										
4 lb ae/gal DGA SL	1.24-1.28 [1.39-1.43]	90-99	4	<0.02	<0.0234	<0.02	<0.0228	0.0200	0.0207	0.00138
4 lb ae/gal DETA SL	1.22-1.28 [1.37-1.43]	90-99	4	<0.02	<0.02	<0.02	<0.02	0.02	0.02	NA
5 lb ae/gal BAPMA SL	1.23-1.26 [1.37-1.42]	90-99	4	<0.02	<0.0206	<0.02	<0.0203	0.02	0.02	0.00015
Corn Stover										
4 lb ae/gal DGA SL	1.24-1.28 [1.39-1.43]	90-99	4	<0.0737	0.453	<0.0881	<0.430	0.157	0.208	0.155
4 lb ae/gal DETA SL	1.22-1.28 [1.37-1.43]	90-99	4	<0.0306	0.680	<0.0318	0.675	0.122	0.238	0.295
5 lb ae/gal BAPMA SL	1.23-1.26 [1.37-1.42]	90-99	4	<0.02	0.667	<0.02	0.616	0.214	0.266	0.251

[1] n = number of field trials.
[2] Values based on total number of samples.
[3] Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation. For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (0.01 ppm). NA = Not applicable.

Soybean

DER Reference: 49379305.der.doc (Magnitude of the Residue in Soybean; Formulation Bridging Study)

The four (4) field trials for field corn were conducted in NAFTA Growing Zone 5 (NE, MN, SD, and IA; 1 trial each). Each treated plot received one preplant soil application at 0.481-0.523 lb acid equivalent (ae)/A (0.539-0.586 kg ae/ha) followed by one foliar broadcast application at 0.965-1.02 lb ae/A (1.08-1.15 kg ae/ha) made at a 119 to 185-day RTI. Applications were made using ground equipment, in spray volumes of ~19-23 gal/A (181-211 L/ha) of water. A NIS was added to spray mixtures for each trial. Samples of soybean forage and hay were harvested at the V6 (BBCH 16) growth stage 66-78 days after the preplant application of dicamba. The registrant indicated that hay was allowed to dry in the field for 2-3 days after harvest. Samples of soybean seed were harvested at a PHI of 7 days after the foliar application. The total application rates achieved for study were 0.481-0.523 lb ae/A (0.539-0.586 kg ae/ha) for soybean forage and hay and 1.45-1.54 lb ae/A (1.63-1.73 kg ae/ha) for seed.

The field residue samples were stored frozen a maximum of 957 days (31.5 months) for forage, 962 days (31.6 months) for hay, and 872 days (28.7 months) for seed from harvest to extraction. Samples were analyzed for the dicamba parent, 5-OH dicamba, and dichlorosalicyclic acid (DCSA) residues of concern within 1-17 days of extraction. Adequate storage stability data are available to show the stability of residues of dicamba and DCSA during frozen storage for up to 4 months in/on soybean forage along with dicamba and 5-OH dicamba for up to 2 years in/on field corn forage, silage, grain, and fodder (D317699, C. Olinger, 12/20/2005). Although the available storage stability data for DCSA do not support the storage durations incurred for this study, its chemical structure is similar to the parent compound and likewise can be concluded to be stable in frozen storage prior to analysis.

The soybean samples were analyzed for residues of dicamba, and the 5-OH dicamba and DCSA metabolites by LC/MS/MS analysis using BASF Method D0902 with minor modifications. The LOQ of the method determined as the LLMV is reported to be 0.01 ppm for each analyte in the soybean seed matrix. An LOQ of 0.05 ppm is reported for each analyte in the soybean forage and hay matrices. The method was verified prior to and in conjunction with the grass sample analysis to demonstrate it was adequate for data collection. Fortification levels for the concurrent method recovery analyses were adequate in bracketing the expected residue levels within an order of magnitude. Quantifiable residues of DCSA and 5-OH dicamba were converted to parent equivalents by the registrant using molecular weight conversion factor of 1.068 and 0.9325, respectively.

Following the side-by-side testing performed to compare the DETA and BAPMA end-use products to the registered DGA formulation, residue levels were found to be similar in soybean RACs (Table 7). At a PHI of 7-days required for the harvesting of soybean seed, average combined residues in this RAC were 1.51 ppm for the DGA salt, 1.77 ppm for the DETA salt, and 0.409 ppm for the BAPMA salt. At the PHI of 66-78 days, average combined residues in soybean forage were 0.15 ppm for the DGA, DETA, and BAPMA salts. For soybean hay collected at a PHI of 74-78 days, combined residues were also <0.15 ppm for the DGA, DETA, and BAPMA salts. No residue decline trials were conducted for soybeans.

Table 7. Summary of Residues from Soybean Field Trials with Dicamba.

Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
				Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Soybean Forage										
4 lb ae/gal DGA SL	0.4807-0.4995 [0.5387-0.5598]	66-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
4 lb ae/gal DETA SL	0.4850-0.5000 [0.5435-0.5604]	66-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
5 lb ae/gal BAPMA SL	0.4873-0.5231 [0.5461-0.5862]	66-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
Soybean Hay										
4 lb ae/gal DGA SL	0.4807-0.4995 [0.5387-0.5598]	74-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
4 lb ae/gal DETA SL	0.4850-0.5000 [0.5435-0.5604]	74-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
5 lb ae/gal BAPMA SL	0.4873-0.5231 [0.5461-0.5862]	74-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
Soybean Seed										
4 lb ae/gal DGA SL	1.4507-1.5227 [1.6296-1.7066]	7	4	<0.03	6.27	<0.03	5.82	0.0937	1.51	2.87

Table 7. Summary of Residues from Soybean Field Trials with Dicamba.										
Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
				Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
4 lb ae/gal DETA SL	1.4583-1.5048 [1.6344-1.6865]	7	4	<0.03	7.00	<0.03	6.84	0.107	1.770	3.38
5 lb ae/gal BAPMA SL	1.4541-1.5403 [1.6296-1.7262]	7	4	<0.03	2.77	<0.03	1.42	0.0922	0.409	0.677

[1] n = number of field trials.
[2] Values based on total number of samples.
[3] Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation. For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (0.05 ppm for forage and hay and 0.01 ppm for seed). NA = Not applicable.

Conclusions: The crop field trial data generated in the bridging studies are supported by adequate storage stability data, concurrent recoveries and a validated data acquisition method. The design of these studies are sufficient in their scope for conducting side-by-side comparison of the new salt formulations of dicamba. It appropriately used a representative crop approach for demonstrating comparable use on numerous crops. The bridging studies were generally made by post-emergence broadcast treatment at the maximum application rate following the minimum PHI using one of the three SL formulations of dicamba. All samples acquired for study were analyzed using an adequate analytical method with the incurred storage intervals appropriately being supported by available stability data. In all, average combined residues of dicamba were found to be similar in/on pasture grass, small grains, field corn, popcorn, and soybean RACs with the results obtained falling well below established tolerance limits. The residue decline trials performed on pasture grass forage and hay show that the combined residues of dicamba will decrease with an increasing PHI. Based on these results, the use of the BASF salt of dicamba on asparagus, proso millet, sorghum, and cotton will be supported as well.

References

DP No.: D375578
Subject: **Dicamba**. Supplemental Residue Field Trial Studies For Dicamba in/on Sweet Corn to Support Petition PP# 0E6209 to Establish Permanent Tolerances.
From: A. Kamel
To: N. Anderson and C. Cyran
Dated: 04/30/2013
MRIDs: 48001303, 48001304, and 48001305

48001303.der.doc Residue Analytical Method – Corn Commodities (DER Reference)
Saha, M. (2009) Method Validation of BASF Analytical Method D0902: "The Determination of Residues of Dicamba (BAS 183 H) and its Metabolite, 5-Hydroxy Dicamba in Corn Matrices using LC/MS/MS". BASF Reg. Doc. No. 2009/7003067. BASF Study Number 357998. Unpublished study prepared by BASF Corporation. 77 p.

DP No.:	D317699
Subject:	Dicamba. Residue Chemistry Considerations for the Reregistration Eligibility Decision (RED) Document. Summary of Analytical Chemistry and Residue Data.
From:	C. Olinger
To:	K. Tyler
Dated:	12/20/2005
MRIDs:	None

B.5.2.1 Analytical Methods for Plant Matrices (Annex IIA 4.3, Annex IIIA 5.3)

B.5.2.1.1 Post-Registration Method (Enforcement)

Document ID:	MRID No. 49379301
Report:	Perez, R. and Perez, S. (2009) Independent Method Validation of BASF Analytical Method D0902: "The Determination of Residues of Dicamba (BAS 183H) and Its Metabolite, 5-Hydroxy Dicamba in Corn Matrices in LC/MS/MS" BASF Study Number 357999. BASF Reg. Doc. No. 2009/7000154. ADPEN Study Number ADPEN-2K9-903-0320. Unpublished study prepared by BASF Crop Protection. 81 p.
Guidelines:	EPA OCSPP Harmonized Test Guideline 860.1340 Residue Analytical Method (August 1996) PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 3 – Residue Analytical Method EU SANCO 825/00/rev. 7 (17/3/04) OECD Guidance Document on Pesticide Residue Analytical Methods
GLP Compliance:	No deviations from regulatory requirements were reported which would have an impact on the validity of the study.
Acceptability:	The study is considered scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document, DP# D429868.
Evaluator:	Peter Savoia, Chemist, Registration Action Branch V/VII/Health Effects Division

Note: This Data Evaluation Record (DER) was originally prepared under contract by Versar, Inc. (6850 Versar Center, Springfield, VA 22151; submitted 12/15/14. The DER has been reviewed by HED and revised to reflect current Office of Pesticide Programs (OPP) policies.

EXECUTIVE SUMMARY

BASF Corporation has submitted the results of an independent laboratory validation (ILV) of a high performance liquid chromatography enforcement method with tandem mass spectrometry detection (LC/MS/MS), BASF Analytical Method D0902, for the determination of residues of dicamba and metabolite 5-hydroxy dicamba (5-OH dicamba) in/on corn matrices. The method is entitled, "BASF Analytical Method D0902: The Determination of Residues of Dicamba (BAS 183 H) and its Metabolite, 5-Hydroxy-Dicamba in Corn Matrices Using LC/MS/MS" and was previously reviewed as a data collection method in conjunction with crop field trial data for sweet corn. Adequate method validation data and descriptions of the method have been submitted previously (see MRID 48001303; DP# 375578, A. Kamel, 4/30/13). The validated limit of quantitation (LOQ; determined as the lowest level of method validation, LLMV) is 0.01 ppm for both analytes in all corn matrices.

The ILV of BASF Method D0902 was conducted by ADPEN Laboratories, Inc. (Jacksonville, FL) using samples of untreated corn grain and stover fortified with dicamba and 5-OH dicamba each at 0.01 and 1.0 ppm (LOQ and 100x LOQ).

The ILV was successful on the first trial; recoveries at both fortification levels for dicamba and 5-OH dicamba were within the acceptable range of 70-120% for corn grain and stover. Contact with the sponsor was not required. The validating laboratory did not identify any critical steps except to make some minor editorial changes to the technical procedures of the method.

The ILV laboratory reported that the method procedure could be completed in 8 hours (1 calendar day) for a set of 12 samples each, without counting the overnight LC/MS/MS analysis time. The method is suitable for additional number of samples, which may be taken through the procedure at one time.

I. Principle of the Method: BASF Analytical Method D0902

BASF Analytical Method D0902 was previously reviewed as a data collection method in conjunction with crop field trial data for sweet corn. Adequate method validation data and descriptions of the method have been submitted previously (see MRID 48001303; DP# 375578, A. Kamel, 4/30/13).

Briefly, homogenized samples are heated with 1 N HCl at ~90 °C for ~45 minutes. The extract is cooled to room temperature and filtered, then adjusted to volume with water. An aliquot of the extract is adjusted to pH 9-10 with concentrated NH_4OH, vortexed, and adjusted to pH 3-4 with concentrated formic acid. Sodium chloride is added, and the extract is partitioned twice with hexane:ethyl acetate (1:1, v:v) followed by centrifugation. The resulting organic phase is reduced to dryness under nitrogen, then reconstituted in methanol:water (10:90, v:v) for analysis by LC/MS/MS. The validated LOQ; determined as the LLMV is 0.01 ppm for both analytes in all corn matrices. The reported limit of detection (LOD) is 0.05 ng/mL, the lowest standard injected.

Table B.5.2.1.1-1. Summary Parameters for the Post-Registration Analytical Method for the Analysis of Dicamba Residues in Corn Grain and Stover.
Not applicable

II. Specificity

The LC/MS/MS method is highly selective for both the quantitation and confirmation of dicamba and 5-OH dicamba. Analysis of control samples resulted in no significant signals at the expected retention times of the analytes. Unambiguous identification is ensured by monitoring two MS/MS transitions characteristic of each analyte as follows:

Dicamba	*m/z* 218.9→174.8 (quantitation) *m/z* 220.9→176.8 (confirmation)
5-OH Dicamba	*m/z* 234.9→154.9 (quantitation) *m/z* 234.9→190.7 (confirmation) *m/z* 236.9→192.7 (confirmation)

The precursor/product ion transitions monitored for dicamba originated from the same ion fragment at *m/z* 218.9 and were 218.9 →174.8 and 220.9→176.8 (the isotope of *m/z* 218.9). However, three different precursor/product ion transitions were monitored for 5-OH dicamba at *m/z* 234.9→154.9, 234.9→190.7, and 236.9→192.7. The first set of transitions is monitored for quantitation purposes, and the second and third set of transitions are monitored for confirmatory purposes. The confirmation ion transition may be used for quantitation if interferences are found with the quantitation ion transition.

During the ILV all transitions were monitored and were available to be used to quantitate residues. The confirmation ion transition (220.9→176.8) was used to quantitate residues of dicamba in corn stover because an interference was observed with the quantitation ion transition (218.9→174.8). Due to interferences observed in both corn grain and stover, the confirmation ion transition (234.9→190.7) was used to quantitate residues for 5-OH dicamba.

The study reviewer notes that the expected retention times used the ILV study for dicamba (10.21 minutes) and 5-OH dicamba (7.73 minutes) differ from the approximate retention times listed in previous review (see MRID 48001303; DP# 375578, A. Kamel, 4/30/13).

The method does not provide for conversion of residues of 5-OH dicamba to parent equivalents. Quantifiable residues may be converted to parent equivalents using molecular weight conversion factor of 0.9325 (MW dicamba 221.04/MW 5-OH dicamba 237.04).

III. Linearity

For each analyte, the linearity of detector response was evaluated using solvent standard solutions. Calibration curves were calculated by linear regression analysis with 1/x weighting. For the least-squares equation, which describes the detector response as a function of the standard concentration, calibration curves resulting from the injection of a minimum of 6 standards over the concentration range of 0.005 to 1.0 ng/mL demonstrated linearity with coefficients of determination (r^2) of at least 0.9986.

IV. Accuracy (Recovery) and Precision (Repeatability)

No radiovalidation data were submitted; however, because the method uses acid hydrolysis procedures to release dicamba and 5-OH dicamba that are similar to those used in the enforcement analytical methods (refer to the Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger), no additional data are required.

Table B.5.2.1.1-2. Radiovalidation of the Post-Registration Analytical Method for Plant Matrices

Matrix	Analyte	Extraction Method	Radioactive Residues (ppm)	Extraction Efficiency[1] (%)
Not applicable				

[1] Extraction efficiency = (residues determined by residue method ÷ residues determined in metabolism study)*100.

BASF method D0902 was previously reviewed as a data collection method in conjunction with crop field trial data for sweet corn and supporting method validation data for corn matrices were submitted and reviewed (48001303.der, A. Kamel, 4/30/13). The method was adequately validated for both sets of ion transitions in samples of sweet corn matrices fortified with a mixed standard of dicamba and 5-OH dicamba at two concentration levels of 0.01(LOQ) and 0.10 ppm.

Table B.5.2.1.1-3. Accuracy and Precision Data for the Validation of BASF Method D0902.						
Analyte	Matrix	Fortification Level (ppm)	Validation Recovery (%)			RSD (%)
			Individual	Mean	Range	
Not applicable						

An ILV of BASF Method D0902 was conducted by ADPEN Laboratories, Inc. (Jacksonville, FL). Samples of untreated sweet corn ears (equates to corn grain) and corn stover (represents corn straw) were sent from BASF to the ILV laboratory. Samples of untreated corn grain and stover were fortified with dicamba and 5-OH dicamba each at 0.01 and 1.0 ppm (LOQ and 100x LOQ). Samples were analyzed using the method as described in Section B.5.2.1.1.

The results of the ILV are presented in Table B.5.2.1.1-4. Contact with the sponsor was not required and acceptable mean recoveries ranging from 77.9-95.1% were obtained for corn grain and stover after the first trial.

The validating laboratory did not identify any critical steps except to make the following editorial recommendations/suggestions:

1. The glassware used for the method should be thoroughly rinsed with water and methanol to prevent contamination.
2. Peak enhancement or suppression could be a potential problem without sufficient sample clean-up. As indicated in the method, it is highly recommended to perform instrument check routinely during LC/MS/MS analysis for standard peak enhancement or suppression. During method development, BASF observed that the response of the analytes could be suppressed due to a heavy load of matrix in the LC/MS/MS analysis. As a result, decreased sensitivity (low signal) of the target analytes and chromatograms with choppy base lines were produced. These problems were observed by BASF by an instrument check sample prior or during the actual sample analysis. The instrument check sample is basically prepared by adding known amount of standard to the control matrix at the LOQ.
3. Matrix interferences were noted for dicamba and 5-OH dicamba, the use of alternate transition ions provided the means to successfully complete the first trial. In the method description, a small note was placed below the tables in Section 3.4.1; however, a clear note should be included in the text of the method to indicate the possibility of interferences and indicate how this would be resolved. Section 7, Potential Problems, would be an appropriate location.
4. The tables provided in Section 3.4.1 Instrumentation and conditions have conflicting instructions with the instructions in Section 3.7.2 Calibration Procedures with respect to the primary and secondary ions to be used. This needs to be resolved.

The ILV laboratory reported that the method procedure could be completed in 8 hours (1 calendar day) for a set of 12 samples each, without counting the overnight LC/MS/MS analysis time. The method is suitable for additional number of samples, which may be taken through the procedure at one time.

Table B.5.2.1.1-4. Accuracy and Precision Data for the Independent Laboratory Validation of BASF Method D0902 for Corn Matrices.						
Analyte	Matrix	Fortification Level (ppm)	Independent Laboratory Validation Recovery (%)			SD (%)
			Individual	Mean	Range	
Dicamba m/z 218.9→174.8 (quantitation)	Corn grain	0.01	96.9, 92.3	91.6	85.7-96.9	4.6
		1.0	85.7, 91.7			
5-OH Dicamba m/z 234.9→190.7 (quantitation)		0.01	100.9, 80.4	95.1	80.4-104.6	10.7
		1.0	94.5, 104.6			
Dicamba m/z 220.9→176.8 (confirmation)	Corn stover	0.01	73.0, 86.7	77.9	73.0-86.7	6.1
		1.0	77.1, 74.8			
5-OH Dicamba m/z 234.9→190.7 (quantitation)		0.01	79.4, 80.6	83.9	79.4-89.3	4.7
		1.0	86.4, 89.3			

V. Limit of Quantitation

Table B.5.2.1.1-5. Summary of Detection and Quantitation Limits for BASF Method D0902.		
Analyte	LOD (ppm)[1]	LOQ (ppm)[2]
Dicamba	0.00005	0.01
5-OH Dicamba	0.00005	0.01

[1] LOD = limit of detection, determined by the lowest standard injected.
[2] LOQ = limit of quantitation, defined as the lowest fortification level where acceptable precision and accuracy data were obtained.

VI. Conclusions

Pending editorial revisions recommended by the independent validation laboratory, adequate recoveries were obtained for corn grain and stover fortified with dicamba and 5-OH dicamba at 0.01 (LOQ) and 1.0 ppm (100x LOQ).

B.5.2.1.2 Pre-Registration Method (Data-Gathering)

BASF method D0902 was previously reviewed as a data collection method in conjunction with crop field trial data for sweet corn and supporting method validation data for corn matrices were submitted and reviewed (48001303.der, A. Kamel, 4/30/13).

B.7.6 Residues Resulting from Supervised Trials (Annex IIA 6.3; Annex IIIA 8.3)

B.7.6.1 Residues in Target Crops

B.7.6.1.4. Pasture Grasses (Bridging data)

Document ID:	MRID No. 49379302
Report:	Norris, F. (2012) Magnitude of the Residue of Dicamba in Pasture Grasses, Formulation Bridging Study. BASF Study Number 389562. BASF Reg. Doc. No. 2012/7004246. Unpublished study prepared by BASF Crop Protection. 382 p.
Guidelines:	EPA OCSPP Harmonized Test Guideline 860.1500 Crop Field Trials (August 1996) PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 9 – Crop Field Trials PMRA Regulatory Directive DIR2010-05 – Revisions to the Residue Chemistry Crop Field Trial Requirements OECD Guideline 509 Crop Field Trial (September 2009)
GLP Compliance:	No deviations from regulatory requirements were reported which would have an impact on the validity of the study.
Acceptability:	The study is considered scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document, DP# D429868.
Evaluator:	Peter Savoia, Chemist, Registration Action Branch V/VII/Health Effects Division

Note: This Data Evaluation Record (DER) was originally prepared under contract by Versar, Inc. (6850 Versar Center, Springfield, VA 22151; submitted 12/15/14. The DER has been reviewed by HED and revised to reflect current Office of Pesticide Programs (OPP) policies.

EXECUTIVE SUMMARY

BASF Crop Protection has submitted field trial data for dicamba on pasture grasses. Four field trials were conducted in the United States during the 2010 growing season in the North American Free Trade Agreement (NAFTA) Growing Zones 3 (FL; 1 trial on Bahiagrass), 5 (MO and NE; 1 trial each on Tall Fescue and Smooth Brome, respectively), and 10 (CA; 1 trial on Bermudagrass). The study was submitted to provide bridging data in support of two new salt formulations of dicamba, the 4 lb ae/gal (480 g ae/L) SL formulation containing diethylenetriamine (DETA) salt (BAS 183 UYH) and the 5 lb ae/gal (600 g ae/L) SL formulation containing N,N-bis-(3-aminopropyl)methylamine (BAPMA) salt (BAS 183 WBH), through side-by-side trials conducted with the 4 lb ae/gal (480 g ae/L) SL formulation containing 2-(2-aminoethoxy)ethanol (a.k.a. diglycolamine; DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137).

Each field site consisted of one untreated plot (Plot 1)and three side-by-side treated plots (Plots 2, 3, and 4) reflecting application of three soluble concentrate (SL) formulations of dicamba each

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containing a different amine salt: the 4 lb ae/gal (480 g ae/L) DGA SL formulation, the 4 lb ae/gal (480 g ae/L) DETA SL formulation, and the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation. Each treated plot received one foliar broadcast application at 0.983-1.02 lb acid equivalent (ae)/A (1.10-1.15 kg ae/ha). Applications were made using ground equipment, in spray volumes of ~20 gal/A (185-193 L/ha) of water. A nonionic surfactant (NIS) was added to spray mixtures for each trial. Samples of grass forage and hay were harvested at PHI of 0, 7, 14, 21, 28/29, and 56 days. Samples of hay were allowed to dry in the field for 2-7 days. In the FL trial, samples of forage from Plot 2 were collected 0-3 days after cutting.

All samples were maintained frozen at the testing facility, during shipping to the laboratory, and were stored frozen until analysis. The field residue samples were stored frozen a maximum of 328 days (10.8 months) for forage and 337 days (11.1 months) for hay from harvest to extraction. Samples were analyzed within 2 days of extraction. Adequate storage stability data are available indicating that residues of dicamba and 5-OH dicamba are stable during frozen storage in/on grass forage and hay for up to 314 and 320 days, respectively (Dicamba RED, DP#317699, 12/20/05, C. Olinger). Adequate storage stability data are therefore available to support the storage conditions and intervals for samples in the current trials.

Samples were analyzed for residues of dicamba and its metabolite 5-OH dicamba using high pressure liquid chromatography with tandem mass spectrometric detection (LC/MS/MS); PSL RA006, which is based upon BASF Method D0902 with minor modifications. The limit of quantitation (LOQ; determined as the lowest limit of method validation, LLMV) was 0.01 ppm for all analytes in all grass matrices. The method was verified prior to and in conjunction with sample analysis and is considered adequate based on acceptable concurrent recovery data. The fortification levels used in concurrent method recovery were adequate to bracket expected residue levels within an order of magnitude. Concurrent recoveries were not corrected for apparent residues in controls; treated samples were not corrected for residues in controls. Quantifiable residues of 5-OH dicamba were converted to parent equivalents by the registrant using molecular weight conversion factor of 0.9325.

The results of the side-by-side trials suggest that combined residues of dicamba and 5-OH dicamba resulting from application of the three SL formulations prepared as different amine salts were similar in pasture grasses. When the trial results are compared on a site-by-site basis, average residues resulting from application of the 4 lb ae/gal (480 g ae/L) DGA SL formulation and the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation were slightly higher than those resulting from the 4 lb ae/gal (480 g ae/L) DETA SL. The maximum mean combined residues of dicamba and 5-OH dicamba in/on grass forage and hay occurred at the 0-day PHI following application of the 5 lb ae/gal (600 g ae/L) SL formulation as the BAPMA salt (BAPMA BAS 183 WBH).

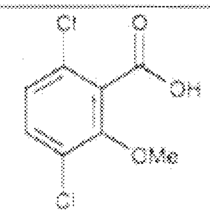
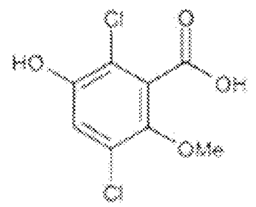
Following a single broadcast foliar application of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a rate of 0.983-1.02 lb ae/A (1.10-1.15 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba in/on **grass forage** were: 37.53-72.24 (38.09-66.34), 38.94-71.25 (44.03-66.51), and 42.94-100.39 (43.28-95.73) ppm, respectively, harvested at a 0-day PHI; 12.37-36.28 (17.73-35.91), 13.16-45.21 (16.31-42.69), and 15.66-37.45 (15.68-36.61) ppm, respectively, harvested at a 7-day PHI; 14.19-44.58 (14.93-42.19), 7.08-33.38 (9.95-30.97), and 10.35-32.17 (11.68-30.07) ppm, respectively, harvested at a 14-day PHI; 12.33-43.31 (12.59-43.00), 7.50-33.36 (7.63-32.25), and 9.17-34.71 (9.44-33.50) ppm, respectively, harvested at a 21-day PHI; 10.11-21.53 (10.38-21.18), 6.76-22.68 (6.76-20.35), and 10.62-20.90 (11.06-20.25) ppm, respectively, harvested at a 28/29-day PHI; and 1.69-5.88 (1.71-4.76), 0.701-5.93 (0.815-5.23), and 0.845-5.79 (0.970-4.50) ppm, respectively, harvested at a 56-day PHI.

Corresponding residues in/on **grass hay** were: 56.46-104.56 (61.74-101.15), 45.05-102.24 (55.36-100.89), and 50.82-118.40 (57.37-111.30) ppm, respectively, harvested at a 0-day PHI; 31.92-50.83 (34.96-46.07), 29.05-57.57 (31.37-54.68), and 33.17-44.84 (34.12-41.76) ppm, respectively, harvested at a 7-day PHI; 31.00-57.49 (32.31-55.91), 18.86-49.70 (22.21-47.10), and 22.39-46.99 (23.64-42.16) ppm, respectively, harvested at a 14-day PHI; 19.94-55.02 (24.52-52.85), 19.32-50.90 (20.00-47.11), and 19.49-53.27 (20.26-47.53) ppm, respectively, harvested at a 21-day PHI; 19.14-35.43 (20.52-31.36), 14.53-25.48 (14.81-25.15), and 12.72-30.71 (12.92-27.96) ppm, respectively, harvested at a 28/29-day PHI; and 3.02-8.83 (3.24-7.64), 2.18-9.84 (2.25-7.13), and 2.76-6.51 (2.82-6.28) ppm, respectively, harvested at a 56-day PHI.

In the residue decline trials, residues of combined dicamba and 5-OH dicamba generally decreased with increasing PHI in/on forage and hay.

I. MATERIALS AND METHODS

A. MATERIALS

Table B.7.6.1.4-1. Nomenclature for Dicamba.	
Common name	Dicamba
Identity	3,6-dichloro-2-methoxybenzoic acid
CAS no.	1918-00-9 (dicamba acid) or 1982-69-0 (sodium salt of dicamba)
Company experimental name	BAS 183 H
Other synonyms (if applicable)	N/A
	
Common name	5-Hydroxy-dicamba
Identity	3,6-dichloro-5-hydroxy-2-methoxy-benzoic acid
CAS no.	7600-50-2
Company experimental name	5-OH dicamba
Other synonyms (if applicable)	N/A
	

B. Study Design

1. Test Procedure

A total of four side-by-side residue trials in/on pasture grasses were conducted with three 4 or 5 lb ae/gal SL formulations during the 2010 growing season. Field trial locations by NAFTA growing zone are summarized in Table B.7.6.1.4.2.

Table B.7.6.1.4-2. Trial Numbers and Geographical Locations.

Crop	No. Trials	NAFTA Growing Zone												Total
		1	2	3	4	5	6	7	8	9	10	11	12	
Grasses	Sub.			1		2					1			4
	Req.[1]													12/9

[1] As per OCSPP 860.1500, Tables 2 and 5, 12 trials, including 4 trials each with Bermudagrass, bluegrass, and bromegrass or fescue, all areas across the country; the second number reflects a 25% reduction in the number of field trials allowed for the crop as a representative commodity in support of a crop group/subgroup tolerance for when application results in no quantifiable residues.

Locations and detailed use patterns for the trials are provided in Table B.7.6.1.4-3.

Table B.7.6.1.4-3. Study Use Pattern.

Location: City, State; Year (Trial ID)	Plot	End-use Product[1]	Method of Application/ Timing of Application	Volume (gal/A) [L/ha]	Rate per Application (lb ae/A) [kg ae/ha]	Retreatment Interval (days)	Total Rate (lb ae/A) [kg ae/ha]	Surfactant/ Adjuvant[2]
Hillsborough, FL; 2010 (R100224)	2	4 lb ae/gal DGA SL	1. Foliar broadcast spray; BBCH 45	19.73 [184.53]	0.9832 [1.102]	--	0.9832 [1.102]	NIS
	3	4 lb ae/gal DETA SL	1. Foliar broadcast spray; BBCH 45	20.02 [187.25]	0.9977 [1.118]	--	0.9977 [1.118]	NIS
	4	5 lb ae/gal BAPMA SL	1. Foliar broadcast spray; BBCH 45	19.98 [186.87]	0.9924 [1.112]	--	0.9924 [1.112]	NIS
Stoddard, MO; 2010 (R100225)	2	4 lb ae/gal DGA SL	1. Foliar broadcast spray; BBCH 33	20.28 [189.68]	1.014 [1.136]	--	1.014 [1.136]	NIS
	3	4 lb ae/gal DETA SL	1. Foliar broadcast spray; BBCH 33	19.96 [186.69]	0.998 [1.118]	--	0.998 [1.118]	NIS
	4	5 lb ae/gal BAPMA SL	1. Foliar broadcast spray; BBCH 33	19.99 [186.97]	0.9995 [1.120]	--	0.9995 [1.120]	NIS
York, NE; 2010 (R100226)	2	4 lb ae/gal DGA SL	1. Foliar broadcast spray; BBCH 32	20.19 [188.84]	1.0006 [1.121]	--	1.0006 [1.121]	NIS
	3	4 lb ae/gal DETA SL	1. Foliar broadcast spray; BBCH 32	20.2 [188.93]	1.0011 [1.122]	--	1.0011 [1.122]	NIS
	4	5 lb ae/gal BAPMA SL	1. Foliar broadcast spray; BBCH 32	20.6 [192.67]	1.0232 [1.146]	--	1.0232 [1.146]	NIS
San Luis Obispo, CA; 2010 (R100227)	2	4 lb ae/gal DGA SL	1. Foliar broadcast spray; BBCH 45	19.9 [186.12]	0.9951 [1.115]	--	0.9951 [1.115]	NIS
	3	4 lb ae/gal DETA SL	1. Foliar broadcast spray; BBCH 45	20.28 [189.68]	1.0141 [1.136]	--	1.0141 [1.136]	NIS
	4	5 lb ae/gal BAPMA SL	1. Foliar broadcast spray; BBCH 45	20.12 [188.18]	1.006 [1.127]	--	1.006 [1.127]	NIS

[1] EP = End-use product; Plot 2: 4 lb ae/gal (480 g ae/L) soluble concentrate (SL) formulation of dicamba as the 2-(2-aminoethoxy)ethanol (DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137); Plot 3: 4 lb ae/gal (480 g ae/L) SL

formulation of dicamba as the diethylenetriamine (DETA) salt (DETA BAS 183 UYH); and Plot 4: 5 lb ac/gal (600 g ae/L) SL formulation as the N,N-Bis-(3-aminopropyl)methylamine (BAPMA) salt (BAPMA BAS 183 WBH).
[2] NIS = nonionic surfactant.

Grass was grown and maintained according to typical agricultural practices. Irrigation was used at the FL and CA trial sites. No unusual weather conditions were reported to have adversely affected crop production or yield during the study.

Sample Handling and Preparation

Single control and duplicate treated samples of grass forage were harvested by hand 0, 7, 14, 21, 28/29, and 56 days after the application. Samples of hay were allowed to dry in the field for 2-7 days. In the FL trial, samples of forage from Plot 2 were collected 0-3 days after cutting. Each RAC sample weighed ≥1 kg. All treated samples were placed in frozen storage at the field trials immediately after harvest. All samples were shipped within 55 days of collection by ACDS freezer truck to BASF Agricultural Research Center (Research Triangle Park, NC) for homogenization, where they were maintained frozen (<-5 °C) prior to preparation. Homogenized samples of grass forage and hay were shipped frozen to the analytical laboratory, Alliance Pharma, Inc. (Malvern, PA) for residue analysis. Grass forage and hay samples were maintained frozen (-20 °C) at the analytical laboratory prior to analysis.

2. Description of Analytical Procedures

Samples of grass (forage and hay) were analyzed for residues of dicamba and 5-OH dicamba using a modified version of LC/MS/MS, which is based on BASF Method D0902. A brief description of the method was included in the submission; for a complete description and method validation refer to 48001303.DER, A. Kamel, 4/30/13.

Briefly, homogenized samples were heated with 1 N HCl at ~90 °C for ~45 minutes. The extract was cooled to room temperature and filtered, then adjusted to volume with water. The extract was adjusted to pH 9-10 with concentrated NH_4OH, vortexed, and adjusted to pH 3-4 with concentrated formic acid. Sodium chloride was added, and the extract was partitioned twice with hexane:ethyl acetate (1:1, v:v) followed by centrifugation. The resulting organic phase was reduced to dryness under nitrogen, then reconstituted in methanol:water (10:90, v:v) for analysis by LC/MS/MS. Two transition ions were monitored for each analyte.

The LOQ (determined as the LLMV), was 0.01 ppm for all analytes in the grass matrices; the corresponding limit of detection (LOD) was 0.004 ppm.

II. RESULTS AND DISCUSSION

Method performance was evaluated by use of concurrent recovery samples. Samples of grass forage and hay were fortified with each analyte at 0.01-100 ppm. Average recoveries were generally within the acceptable range of 70-120%. The method was considered valid for the analysis of dicamba residues in/on grass matrices (Table B.7.6.1.4-4). The fortification levels bracketed the measured residues within an order of magnitude. Concurrent recoveries were not corrected for apparent residues in controls; residues in the treated samples were not corrected for apparent residues in controls.

For analysis of grass forage and hay, the detector response was linear (coefficient of determination, $r^2 \geq 0.9807$ for dicamba and $r^2 \geq 0.9892$ for 5-OH dicamba within the range of 0.1-50 ng/mL). Representative chromatograms of control samples, fortified samples, and treated samples were provided. The control chromatograms generally had no peaks of interest above the chromatographic background. The fortified sample chromatograms contained only the analyte of interest, and peaks were symmetrical and well defined. Apparent residues of each analyte in controls were <0.01 ppm with the exception of two samples each of forage and hay which bore detectable residues of dicamba at 0.011-0.036 and 0.061-0.085 ppm, respectively. Corresponding residues of 5-OH dicamba in these samples were <0.01 ppm. The reported residue values were not corrected for apparent residues in controls. Residues of 5-OH dicamba are expressed in parent equivalents as calculated by the registrant using a molecular weight conversion factor of 0.9325.

Table B.7.6.1.4-4. Summary of Procedural/Concurrent Recoveries of Dicamba and 5-OH Dicamba from Grass Matrices.

Matrix	Analyte	Fortification Level (ppm)	Sample size (n)	Recoveries[1] (%)	Mean ± Std. Dev. (%)
Forage	Dicamba	0.01-100	12	85.7-111.6	98.2 ± 7.8
	5-OH Dicamba	0.01-100	12	84.5-107.4; 120.9; 127.9	98.9 ± 13.5
Hay	Dicamba	0.01-100	12	75.2-108.6	92.8 ± 10.0
	5-OH Dicamba	0.01-100	12	75.6-99.6	91.9 ± 8.0

[1] Concurrent recoveries were not corrected for apparent residues in controls because residues were nondetectable in the associated controls.

The field residue samples were stored frozen a maximum of 328 days (10.8 months) for forage and 337 days (11.1 months) for hay from harvest to extraction (Table B.7.6.1.4-5). Samples were analyzed within 2 days of extraction. Adequate storage stability data are available indicating that residues of dicamba and 5-OH dicamba are stable during frozen storage in/on grass forage and hay for up to 314 and 320 days, respectively (Dicamba RED, DP#317699, 12/20/05, C. Olinger). These data are acceptable to support the storage conditions and durations of samples from the submitted field trials.

Table B.7.6.1.4-5. Summary of Storage Conditions.			
Matrix	Storage Temperature (°C)	Actual Storage Duration[1]	Interval of Demonstrated Storage Stability
Forage	≤ -10 at the field sites; ≤ -5 at BASF; and ≤ -20 at the analytical laboratory	128-328 days (4.2-10.8 months)	Adequate storage stability data are available indicating that residues of dicamba and 5-OH dicamba are stable under frozen storage in/on grass forage and hay for up to 314 and 320 days, respectively.[2]
Hay		136-337 days (4.5-11.1 months)	

[1] Interval from harvest to extraction. Samples were analyzed within 2 days of extraction.
[2] Dicamba RED, DP#317699, 12/20/05, C. Olinger.

The results from the submitted field trials are presented in Tables B.7.6.1.4-6 and B.7.6.1.4-7. The results of the side-by-side trials suggest that combined residues of dicamba and 5-OH dicamba resulting from application of the three SL formulations prepared as different amine salts were similar in pasture grasses. When the trial results are compared on a site-by-site basis, average residues resulting from application of the 4 lb ae/gal (480 g ae/L) DGA SL formulation and the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation were slightly higher than those resulting from the 4 lb ae/gal (480 g ae/L) DETA SL. The maximum mean combined residues of dicamba and 5-OH dicamba in/on grass forage and hay occurred at the 0-day PHI following application of the 5 lb ae/gal (600 g ae/L) SL formulation as the BAPMA salt (BAPMA BAS 183 WBH).

Following a single broadcast foliar application of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a rate of 0.983-1.02 lb ae/A (1.10-1.15 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba in/on **grass forage** were: 37.53-72.24 (38.09-66.34), 38.94-71.25 (44.03-66.51), and 42.94-100.39 (43.28-95.73) ppm, respectively, harvested at a 0-day PHI; 12.37-36.28 (17.73-35.91), 13.16-45.21 (16.31-42.69), and 15.66-37.45 (15.68-36.61) ppm, respectively, harvested at a 7-day PHI; 14.19-44.58 (14.93-42.19), 7.08-33.38 (9.95-30.97), and 10.35-32.17 (11.68-30.07) ppm, respectively, harvested at a 14-day PHI; 12.33-43.31 (12.59-43.00), 7.50-33.36 (7.63-32.25), and 9.17-34.71 (9.44-33.50) ppm, respectively, harvested at a 21-day PHI; 10.11-21.53 (10.38-21.18), 6.76-22.68 (6.76-20.35), and 10.62-20.90 (11.06-20.25) ppm, respectively, harvested at a 28/29-day PHI; and 1.69-5.88 (1.71-4.76), 0.701-5.93 (0.815-5.23), and 0.845-5.79 (0.970-4.50) ppm, respectively, harvested at a 56-day PHI.

Corresponding residues in/on **grass hay** were: 56.46-104.56 (61.74-101.15), 45.05-102.24 (55.36-100.89), and 50.82-118.40 (57.37-111.30) ppm, respectively, harvested at a 0-day PHI; 31.92-50.83 (34.96-46.07), 29.05-57.57 (31.37-54.68), and 33.17-44.84 (34.12-41.76) ppm, respectively, harvested at a 7-day PHI; 31.00-57.49 (32.31-55.91), 18.86-49.70 (22.21-47.10), and 22.39-46.99 (23.64-42.16) ppm, respectively, harvested at a 14-day PHI; 19.94-55.02 (24.52-52.85), 19.32-50.90 (20.00-47.11), and 19.49-53.27 (20.26-47.53) ppm, respectively, harvested at a 21-day PHI; 19.14-35.43 (20.52-31.36), 14.53-25.48 (14.81-25.15), and 12.72-30.71 (12.92-27.96) ppm, respectively, harvested at a 28/29-day PHI; and 3.02-8.83 (3.24-7.64), 2.18-9.84 (2.25-7.13), and 2.76-6.51 (2.82-6.28) ppm, respectively, harvested at a 56-day PHI.

In the residue decline trials, residues of combined dicamba and 5-OH dicamba generally decreased with increasing PHI in/on forage and hay.

Table B.7.6.1.4-6. Residue Data from Grass Field Trials with Dicamba.[1]									
Location: City, State: Year (Trial ID)	Zone	Grass/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
Hillsborough, FL; 2010 (R100224)	3	Bahia	2	Forage	0.983 [1.10]	0 (2)	59.71, 71.25 [65.48]	0.737, 0.993 [0.865]	60.44, 72.24 [66.34]
						7	24.76, 22.78 [23.77]	2.01, 1.90 [1.96]	26.77, 24.68 [25.73]
						14 (2)	38.29, 35.13 [36.71]	6.29, 4.66 [5.48]	44.58, 39.80 [42.19]
						21 (2)	33.83, 27.93 [30.88]	5.61, 4.25 [4.93]	39.45, 32.17 [35.81]
						28 (3)	13.43, 9.03 [11.23]	3.45, 2.60 [3.03]	16.89, 11.63 [14.26]
						56	3.23, 2.94 [3.08]	0.863, 0.824 [0.844]	4.09, 3.76 [3.93]
				Hay		0 (4)	76.90, 75.91 [76.41]	1.16, 1.12 [1.14]	78.07, 77.03 [77.55]
						7 (2)	44.19, 28.73 [36.46]	3.85, 3.18 [3.52]	48.04, 31.92 [39.98]
						14 (3)	29.17, 45.98 [37.58]	4.19, 7.14 [5.67]	33.37, 53.12 [43.24]
						21 (4)	40.02, 29.02 [34.52]	6.05, 4.58 [5.31]	46.07, 33.60 [39.83]
						28 (5)	26.17, 19.81 [22.99]	4.62, 3.55 [4.09]	30.79, 23.37 [27.08]
						56 (4)	5.20, 4.00 [4.60]	1.87, 1.33 [1.60]	7.07, 5.32 [6.20]
			3	Forage	0.998 [1.12]	0	50.81, 57.83 [54.32]	0.582, 0.574 [0.578]	51.39, 58.41 [54.90]
						7	27.41, 25.76 [26.59]	1.97, 2.18 [2.07]	29.38, 27.94 [28.66]
						14	25.65, 27.64 [26.64]	3.68, 4.54 [4.11]	29.33, 32.18 [30.75]
						21	28.96, 26.84 [27.90]	4.39, 4.29 [4.34]	33.36, 31.14 [32.25]
						28	14.71, 12.64 [13.67]	3.83, 3.22 [3.53]	18.54, 15.86 [17.20]
						56	2.87, 3.17 [3.02]	0.704, 0.796 [0.750]	3.58, 3.96 [3.77]
				Hay		0 (4)	62.15, 57.33 [59.74]	0.769, 0.622 [0.696]	62.92, 57.95 [60.43]
						7 (2)	41.67, 31.54 [36.61]	3.69, 3.15 [3.42]	45.36, 34.69 [40.03]
						14 (3)	32.68, 25.09 [28.89]	4.33, 3.30 [3.81]	37.01, 28.39 [32.70]
						21 (4)	22.08, 35.14 [28.61]	3.56, 4.99 [4.28]	25.64, 40.13 [32.89]
						28 (5)	16.58, 15.91 [16.25]	3.34, 3.06 [3.20]	19.92, 18.97 [19.44]
						56 (4)	4.43, 4.57 [4.50]	1.58, 1.45 [1.51]	6.01, 6.02 [6.01]

Table B.7.6.1.4-6. Residue Data from Grass Field Trials with Dicamba.[1]

Location: City, State: Year (Trial ID)	Zone	Grass/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
Hillsborough, FL: 2010 (R100224), *continued*	3	Bahia	4	Forage	0.992 [1.11]	0	56.08, 57.20 [56.64]	0.972, 0.822 [0.897]	57.05, 58.02 [57.53]
						7	34.17, 32.79 [33.48]	3.28, 2.97 [3.12]	37.45, 35.76 [36.61]
						14	16.07, 22.51 [19.29]	3.07, 4.56 [3.82]	19.14, 27.08 [23.11]
						21	25.12, 24.48 [24.80]	4.50, 5.24 [4.87]	29.62, 29.72 [29.67]
						28	12.60, 8.97 [10.78]	4.59, 3.73 [4.16]	17.19, 12.69 [14.94]
						56	3.00, 3.16 [3.08]	0.909, 1.03 [0.968]	3.91, 4.19 [4.05]
				Hay		0 (4)	50.22, 63.12 [56.67]	0.599, 0.815 [0.707]	50.82, 63.93 [57.37]
						7 (2)	40.71, 33.61 [37.16]	4.13, 3.10 [3.61]	44.84, 36.71 [40.77]
						14 (3)	25.38, 26.05 [25.71]	5.23, 5.42 [5.33]	30.60, 31.47 [31.04]
						21 (4)	22.38, 33.22 [27.80]	4.22, 6.56 [5.39]	26.60, 39.77 [33.18]
						28 (5)	23.71, 15.44 [19.57]	5.38, 3.97 [4.67]	29.09, 19.41 [24.25]
						56 (4)	4.67, 4.27 [4.47]	1.84, 1.78 [1.81]	6.51, 6.04 [6.28]
Stoddard, MO; 2010 (R100225)	5	Tall fescue	2	Forage	1.01 [1.14]	0	37.51, 38.60 [38.05]	0.030, 0.038 [0.034]	37.54, 38.64 [38.09]
						7	33.79, 33.70 [33.74]	2.50, 1.85 [2.17]	36.28, 35.54 [35.91]
						14	37.76, 33.42 [35.59]	2.40, 1.94 [2.17]	40.15, 35.36 [37.76]
						21	39.27, 40.38 [39.82]	3.42, 2.93 [3.17]	42.69, 43.31 [43.00]
						29	18.06, 16.82 [17.44]	3.47, 4.00 [3.73]	21.53, 20.82 [21.18]
						56	2.19, 2.18 [2.18]	1.94, 1.71 [1.82]	4.13, 3.88 [4.00]
				Hay		0 (3)	55.33, 65.77 [60.55]	1.13, 1.24 [1.19]	56.46, 67.02 [61.74]
						7 (3)	44.36, 37.20 [40.78]	2.51, 2.20 [2.36]	46.87, 39.40 [43.13]
						14 (3)	51.02, 53.52 [52.27]	3.31, 3.97 [3.64]	54.33, 57.49 [55.91]
						21 (3)	47.17, 51.56 [49.36]	3.51, 3.46 [3.49]	50.69, 55.02 [52.85]
						29 (4)	24.43, 31.69 [28.06]	2.87, 3.74 [3.31]	27.30, 35.43 [31.36]
						56 (5)	4.79, 3.50 [4.14]	1.91, 1.32 [1.61]	6.69, 4.81 [5.75]

Table B.7.6.1.4-6. Residue Data from Grass Field Trials with Dicamba.[1]									
Location: City, State; Year (Trial ID)	Zone	Grass/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
Stoddard, MO; 2010 (R100225), *continued*	5	Tall fescue	3	Forage	0.998 [1.12]	0	38.88, 49.05 [43.97]	0.061, 0.069 [0.065]	38.94, 49.12 [44.03]
						7	38.77, 42.68 [40.72]	1.39, 2.54 [1.96]	40.16, 45.21 [42.69]
						14	26.70, 31.16 [28.93]	1.87, 2.22 [2.04]	28.57, 33.38 [30.97]
						21	31.58, 28.99 [30.29]	1.38, 1.61 [1.49]	32.96, 30.60 [31.78]
						29	20.18, 15.55 [17.86]	2.49, 2.48 [2.48]	22.68, 18.02 [20.35]
						56	3.27, 5.05 [4.16]	1.26, 0.881 [1.07]	4.53, 5.93 [5.23]
				Hay		0 (3)	64.57, 44.20 [54.39]	1.11, 0.850 [0.978]	65.68, 45.05 [55.36]
						7 (3)	49.08, 54.39 [51.73]	2.71, 3.18 [2.95]	51.79, 57.57 [54.68]
						14 (3)	47.32, 41.45 [44.38]	2.38, 3.06 [2.72]	49.70, 44.50 [47.10]
						21 (3)	38.05, 48.18 [43.11]	5.28, 2.73 [4.00]	43.33, 50.90 [47.11]
						29 (4)	22.60, 21.57 [22.09]	2.88, 3.24 [3.06]	25.48, 24.81 [25.15]
						56 (5)	8.36, 3.83 [6.10]	1.47, 0.591 [1.03]	9.84, 4.43 [7.13]
			4	Forage	1.00 [1.12]	0	49.92, 43.60 [43.26]	0.021, 0.018 [0.020]	42.94, 43.62 [43.28]
						7	35.94, 33.32 [34.63]	1.05, 1.03 [1.04]	36.98, 34.35 [35.67]
						14	26.35, 30.33 [28.34]	1.61, 1.84 [1.73]	27.96, 32.17 [30.07]
						21	32.65, 30.75 [31.70]	2.06, 1.54 [1.80]	34.71, 32.29 [33.50]
						29	15.99, 14.20 [15.09]	1.04, 0.877 [0.957]	17.02, 15.07 [16.05]
						56	2.97, 5.46 [4.22]	0.248, 0.328 [0.288]	3.22, 5.79 [4.50]
				Hay		0 (3)	64.81, 63.99 [64.40]	0.615, 0.808 [0.712]	65.43, 64.80 [65.11]
						7 (3)	35.65, 41.24 [38.44]	2.11, 2.54 [2.33]	37.76, 43.77 [40.77]
						14 (3)	45.40, 36.19 [40.79]	1.59, 1.14 [1.37]	46.99, 37.34 [42.16]
						21 (3)	50.93, 40.18 [45.56]	2.34, 1.61 [1.98]	53.27, 41.79 [47.53]
						29 (4)	29.29, 23.86 [26.57]	1.42, 1.36 [1.39]	30.71, 25.21 [27.96]
						56 (5)	5.47, 4.70 [5.08]	0.443, 0.406 [0.425]	5.91, 5.10 [5.51]

Table B.7.6.1.4-6. Residue Data from Grass Field Trials with Dicamba.[1]									
Location: City, State; Year (Trial ID)	Zone	Grass/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
York, NE; 2010 (R100226)	5	Smooth brome	2	Forage	1.00 [1.12]	0	45.22, 54.74 [49.98]	1.35, 1.11 [1.23]	46.57, 55.85 [51.21]
						7	6.94, 3.05 [5.00]	16.16, 9.32 [12.74]	23.10, 12.37 [17.73]
						14	2.03, 2.28 [2.15]	12.17, 13.38 [12.77]	14.19, 15.66 [14.93]
						21	1.10, 0.837 [0.967]	13.02, 11.49 [12.26]	14.12, 12.33 [13.22]
						28	0.445, 0.369 [0.407]	10.44, 9.97 [10.21]	10.88, 10.34 [10.61]
						56	0.067, 0.073 [0.070]	3.58, 5.81 [4.69]	3.65, 5.88 [4.76]
				Hay		0 (7)	93.35, 89.41 [91.38]	11.21, 8.35 [9.78]	104.56, 97.75 [101.15]
						7 (4)	10.87, 7.82 [9.35]	39.96, 33.49 [36.73]	50.83, 41.31 [46.07]
						14 (7)	4.41, 3.77 [4.09]	28.51, 27.94 [28.22]	32.91, 31.71 [32.31]
						21 (7)	1.33, 1.87 [1.60]	18.61, 27.24 [22.92]	19.94, 29.11 [24.52]
						28 (3)	0.834, 0.911 [0.873]	22.58, 24.64 [23.61]	23.41, 25.55 [24.48]
						56 (2)	0.110, 0.093 [0.102]	8.72, 6.36 [7.54]	8.83, 6.46 [7.64]
			3	Forage	1.00 [1.12]	0	46.46, 51.26 [48.86]	1.96, 1.62 [1.79]	48.42, 52.89 [50.65]
						7	3.91, 5.49 [4.70]	9.25, 13.97 [11.61]	13.16, 19.46 [16.31]
						14	1.01, 1.73 [1.37]	6.07, 11.08 [8.58]	7.08, 12.81 [9.95]
						21	0.952, 0.723 [0.838]	7.18, 6.95 [7.06]	8.13, 7.67 [7.90]
						28	0.414, 0.341 [0.378]	7.08, 6.94 [7.01]	7.50, 7.28 [7.39]
						56	0.053, 0.049 [0.051]	1.97, 1.92 [1.94]	2.02, 1.97 [1.99]
				Hay		0 (7)	90.53, 90.88 [90.71]	9.01, 11.36 [10.18]	99.54, 102.24 [100.89]
						7 (4)	5.57, 7.51 [6.54]	23.48, 28.51 [26.00]	29.05, 36.02 [32.53]
						14 (7)	2.73, 3.24 [2.98]	16.13, 22.31 [19.22]	18.86, 25.55 [22.21]
						21 (7)	1.82, 1.58 [1.70]	17.50, 19.11 [18.30]	19.32, 20.69 [20.00]
						28 (3)	0.731, 0.725 [0.728]	15.31, 16.03 [15.67]	16.05, 16.75 [16.40]
						56 (2)	0.083, 0.114 [0.099]	3.83, 4.18 [4.01]	3.92, 4.30 [4.11]

Table B.7.6.1.4-6. Residue Data from Grass Field Trials with Dicamba.[1]									
Location: City, State; Year (Trial ID)	Zone	Grass/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI[2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
York, NE; 2010 (R100226), *continued*	5	Smooth brome	4	Forage	1.02 [1.15]	0	48.96, 46.01 [47.48]	0.634, 1.04 [0.839]	49.59, 47.06 [48.32]
						7	3.77, 4.34 [4.06]	11.89, 11.37 [11.63]	15.66, 15.71 [15.68]
						14	1.52, 1.73 [1.62]	8.83, 11.27 [10.05]	10.35, 13.01 [11.68]
						21	0.690, 0.593 [0.642]	9.03, 8.57 [8.80]	9.72, 9.17 [9.44]
						28	1.61, 1.60 [1.61]	9.00, 9.91 [9.46]	10.62, 11.50 [11.06]
						56	0.060, 0.062 [0.061]	2.30, 2.48 [2.39]	2.36, 2.54 [2.45]
				Hay		0[6] (7)	93.03, 108.15 [100.59]	11.18, 10.26 [10.72]	104.20, 118.40 [111.30]
						7 (4)	6.35, 6.58 [6.47]	26.82, 28.49 [27.66]	33.17, 35.07 [34.12]
						14 (7)	2.43, 2.73 [2.58]	19.96, 22.17 [21.07]	22.39, 24.90 [23.64]
						21 (7)	1.14, 1.24 [1.19]	18.35, 19.78 [19.07]	19.49, 21.02 [20.26]
						28[6] (3)	0.634, 0.422 [0.528]	21.66, 17.42 [19.54]	22.29, 17.85 [20.07]
						56 (2)	0.094, 0.089 [0.092]	4.73, 3.57 [4.15]	4.83, 3.66 [4.24]
San Luis Obispo, CA; 2010 (R100227)	10	Bermuda	2	Forage	0.995 [1.12]	0	46.61, 42.73 [44.67]	0.098, 0.072 [0.085]	46.71, 42.80 [44.76]
						7	20.54, 15.10 [17.82]	4.95, 3.73 [4.34]	25.49, 18.83 [22.16]
						14	15.10, 14.17 [14.63]	4.83, 4.38 [4.61]	19.93, 18.55 [19.24]
						21	9.48, 9.44 [9.46]	3.27, 2.98 [3.13]	12.76, 12.42 [12.59]
						28	6.52, 6.92 [6.72]	3.59, 3.73 [3.66]	10.11, 10.65 [10.38]
						56	1.20, 1.25 [1.22]	0.490, 0.492 [0.491]	1.69, 1.74 [1.71]
				Hay		0 (3)	60.32, 62.15 [61.24]	3.19, 2.09 [2.64]	63.51, 64.24 [63.87]
						7 (3)	28.26, 26.18 [27.22]	8.02, 7.45 [7.73]	36.28, 33.63 [34.96]
						14 (3)	24.57, 28.08 [26.33]	6.43, 7.84 [7.14]	31.00, 35.92 [33.46]
						21 (3)	21.14, 22.21 [22.68]	7.42, 7.99 [7.70]	30.56, 30.20 [30.38]
						28 (3)	15.58, 13.32 [14.45]	6.31, 5.82 [6.07]	21.89, 19.14 [20.52]
						56 (3)	2.35, 2.04 [2.19]	1.11, 0.986 [1.05]	3.47, 3.02 [3.24]

Table B.7.6.1.4-6. Residue Data from Grass Field Trials with Dicamba.[1]

Location: City, State; Year (Trial ID)	Zone	Grass/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average] Dicamba	5-OH Dicamba[4]	Combined[5]
San Luis Obispo, CA; 2010 (R100227), *continued*	10	Bermuda	3	Forage	1.01 [1.14]	0	61.61, 71.15 [66.38]	0.151, 0.108 [0.130]	61.76, 71.25 [66.51]
						7	17.12, 16.44 [16.78]	5.71, 4.80 [5.26]	22.84, 21.24 [22.04]
						14	10.71, 12.35 [11.53]	4.90, 6.08 [5.49]	15.61, 18.43 [17.02]
						21	5.00, 4.81 [4.90]	2.76, 2.69 [2.72]	7.76, 7.50 [7.63]
						28	4.52, 4.47 [4.50]	2.23, 2.30 [2.27]	6.76, 6.77 [6.76]
						56	0.617, 0.482 [0.550]	0.312, 0.219 [0.266]	0.929, 0.701 [0.815]
				Hay		0 (3)	77.69, 79.63 [78.66]	3.25, 2.60 [2.93]	80.94, 82.24 [81.59]
						7 (3)	22.28, 24.09 [23.19]	8.38, 7.98 [8.18]	30.66, 32.07 [31.37]
						14 (3)	19.75, 21.45 [20.60]	7.29, 6.89 [7.09]	27.03, 28.33 [27.68]
						21 (3)	14.02, 15.79 [14.91]	8.12, 9.32 [8.72]	22.15, 25.11 [23.63]
						28 (3)	10.64, 11.18 [10.91]	3.89, 3.92 [3.91]	14.53, 15.09 [14.81]
						56 (3)	1.62, 1.57 [1.59]	0.704, 0.612 [0.658]	2.32, 2.18 [2.25]
			4	Forage	1.01 [1.13]	0	100.02, 90.73 [95.38]	0.368, 0.345 [0.357]	100.39, 91.08 [95.73]
						7	28.80, 30.13 [29.46]	4.75, 5.40 [5.07]	33.54, 35.53 [34.54]
						14	13.07, 13.05 [13.06]	8.19, 5.78 [6.99]	21.26, 18.83 [20.04]
						21	9.94, 8.05 [9.00]	6.59, 4.68 [5.63]	16.53, 12.73 [14.63]
						28	12.75, 13.35 [13.05]	8.14, 6.26 [7.20]	20.90, 19.60 [20.25]
						56	0.638, 0.481 [0.560]	0.457, 0.364 [0.411]	1.10, 0.845 [0.970]
				Hay		0 (3)	87.24, 97.66 [92.45]	10.14, 7.93 [9.04]	97.38, 105.58 [101.48]
						7 (3)	37.12, 34.52 [35.82]	6.22, 5.66 [5.94]	43.33, 40.19 [41.76]
						14 (3)	25.99, 24.82 [25.41]	10.74, 10.04 [10.39]	36.73, 34.86 [35.80]
						21 (3)	19.18, 18.12 [18.65]	14.20, 10.93 [12.57]	33.38, 29.05 [31.22]
						28 (3)	8.43, 8.72 [8.57]	4.29, 4.41 [4.35]	12.72, 13.13 [12.92]
						56 (3)	1.30, 1.29 [1.30]	1.46, 1.58 [1.52]	2.76, 2.88 [2.82]

[1] End-use product: Plot 2: 4 lb ae/gal (480 g ae/L) soluble concentrate (SL) formulation of dicamba as the 2-(2-aminoethoxy)ethanol (DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137); Plot 3: 4 lb ae/gal (480 g ae/L) SL formulation of dicamba as the diethylenetriamine (DETA) salt (DETA BAS 183 UYH); and Plot 4: 5 lb ae/gal (600 g ae/L) SL formulation as the N,N-Bis-(3-aminopropyl)methylamine (BAPMA) salt (BAPMA BAS 183 WBH).

[2] Days after application. The number of days samples were allowed to dry between harvest and collection is reported in parentheses.
[3] The LOD was 0.004 ppm, and the LOQ was 0.01 ppm for each analyte. For samples with nonquantifiable residues, combined residues and per trial averages were calculated by the study reviewer using the LOQ for residues <LOQ. For samples with quantifiable residues, combined residues and per trial averages were calculated by the registrant.
[4] Residues of 5-OH dicamba are expressed in parent equivalents as calculated by the registrant using a molecular weight conversion factor of 0.9325.
[5] Combined residues are the sum of dicamba and 5-OH dicamba.
[6] The registrant stated that a review of the data from the analytical report indicates that one 0-day hay sample and one 28-day hay sample were reversed

Table B.7.6.1.4-7. Summary of Residues from Grass Field Trials with Dicamba.

Crop Matrix	Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
					Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Grass, forage	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	0	4	37.53	72.24	38.09	66.34	47.98	50.10	12.08
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	38.94	71.25	44.03	66.51	52.77	54.02	9.45
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	42.94	100.39	43.28	95.73	52.93	61.22	23.75
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	7	4	12.37	36.28	17.73	35.91	23.94	25.38	7.74
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	13.16	45.21	16.31	42.69	25.35	27.42	11.36
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	15.66	37.45	15.68	36.61	35.10	30.62	10.00
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	14	4	14.19	44.58	14.93	42.19	28.50	28.53	13.45
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	7.08	33.38	9.95	30.97	23.89	22.17	10.44
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	10.35	32.17	11.68	30.07	21.58	21.22	7.62
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	21	4	12.33	43.31	12.59	43.00	24.52	26.12	15.58
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	7.50	33.36	7.63	32.25	19.84	19.89	14.00
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	9.17	34.71	9.44	33.50	22.15	21.81	11.59
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	28/29	4	10.11	21.53	10.38	21.18	12.44	14.11	5.04
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	6.76	22.68	6.76	20.35	12.30	12.93	6.88
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	10.62	20.90	11.06	20.25	15.50	15.58	3.78
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	56	4	1.69	5.88	1.71	4.76	3.96	3.60	1.31
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	0.701	5.93	0.815	5.23	2.88	2.95	1.94
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	0.845	5.79	0.970	4.50	3.25	2.99	1.61

Table B.7.6.1.4-7. Summary of Residues from Grass Field Trials with Dicamba.

Crop Matrix	Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
					Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Grass, hay	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	0	4	56.46	104.56	61.74	101.15	70.71	76.08	18.12
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	45.05	102.24	55.36	100.89	71.01	74.57	20.90
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	50.82	118.40	57.37	111.30	83.30	83.82	26.56
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	7	4	31.92	50.83	34.96	46.07	41.55	41.03	4.76
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	29.05	57.57	31.37	54.68	36.28	39.65	10.73
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	33.17	44.84	34.12	41.76	40.77	39.36	3.52
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	14	4	31.00	57.49	32.31	55.91	38.35	41.23	10.94
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	18.86	49.70	22.21	47.10	30.19	32.42	10.68
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	22.39	46.99	23.64	42.16	33.42	33.16	7.81
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	21	4	19.94	55.02	24.52	52.85	35.11	36.90	12.37
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	19.32	50.90	20.00	47.11	28.26	30.91	12.09
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	19.49	53.27	20.26	47.53	32.20	33.05	11.21
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	28/29	4	19.14	35.43	20.52	31.36	25.78	25.86	4.55
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	14.53	25.48	14.81	25.15	17.92	18.95	4.56
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	12.72	30.71	12.92	27.96	22.16	21.30	6.45
	4 lb ae/gal DGA SL	0.983-1.01 [1.10-1.14]	56	4	3.02	8.83	3.24	7.64	5.97	5.71	1.83
	4 lb ae/gal DETA SL	0.998-1.01 [1.12-1.14]		4	2.18	9.84	2.25	7.13	5.06	4.87	2.15
	5 lb ae/gal BAPMA SL	0.992-1.02 [1.11-1.15]		4	2.76	6.51	2.82	6.28	4.87	4.71	1.52

[1] n = number of field trials.
[2] Values based on total number of samples.
[3] Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation.

III. CONCLUSIONS

The pasture grass field trials are considered scientifically acceptable. The results of the side-by-side trials suggest that combined residues of dicamba and 5-OH dicamba resulting from application of the three SL formulations prepared as different amine salts were similar in pasture grasses. When the trial results are compared on a site-by-site basis, average residues resulting from application of the 4 lb ae/gal (480 g ae/L) DGA SL formulation and the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation were slightly higher than those resulting from the 4 lb ae/gal (480 g ae/L) DETA SL. The maximum mean combined residues of dicamba and 5-OH dicamba in/on grass forage and hay occurred at the 0-day PHI following application of the 5 lb ae/gal (600 g ae/L) SL formulation as the BAPMA salt (BAPMA BAS 183 WBH).

Following a single broadcast foliar application of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a rate of 0.983-1.02 lb ae/A (1.10-1.15 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba in/on **grass forage** were: 37.53-72.24 (38.09-66.34), 38.94-71.25 (44.03-66.51), and 42.94-100.39 (43.28-95.73) ppm, respectively, harvested at a 0-day PHI; 12.37-36.28 (17.73-35.91), 13.16-45.21 (16.31-42.69), and 15.66-37.45 (15.68-36.61) ppm, respectively, harvested at a 7-day PHI; 14.19-44.58 (14.93-42.19), 7.08-33.38 (9.95-30.97), and 10.35-32.17 (11.68-30.07) ppm, respectively, harvested at a 14-day PHI; 12.33-43.31 (12.59-43.00), 7.50-33.36 (7.63-32.25), and 9.17-34.71 (9.44-33.50) ppm, respectively, harvested at a 21-day PHI; 10.11-21.53 (10.38-21.18), 6.76-22.68 (6.76-20.35), and 10.62-20.90 (11.06-20.25) ppm, respectively, harvested at a 28/29-day PHI; and 1.69-5.88 (1.71-4.76), 0.701-5.93 (0.815-5.23), and 0.845-5.79 (0.970-4.50) ppm, respectively, harvested at a 56-day PHI.

Corresponding residues in/on **grass hay** were: 56.46-104.56 (61.74-101.15), 45.05-102.24 (55.36-100.89), and 50.82-118.40 (57.37-111.30) ppm, respectively, harvested at a 0-day PHI; 31.92-50.83 (34.96-46.07), 29.05-57.57 (31.37-54.68), and 33.17-44.84 (34.12-41.76) ppm, respectively, harvested at a 7-day PHI; 31.00-57.49 (32.31-55.91), 18.86-49.70 (22.21-47.10), and 22.39-46.99 (23.64-42.16) ppm, respectively, harvested at a 14-day PHI; 19.94-55.02 (24.52-52.85), 19.32-50.90 (20.00-47.11), and 19.49-53.27 (20.26-47.53) ppm, respectively, harvested at a 21-day PHI; 19.14-35.43 (20.52-31.36), 14.53-25.48 (14.81-25.15), and 12.72-30.71 (12.92-27.96) ppm, respectively, harvested at a 28/29-day PHI; and 3.02-8.83 (3.24-7.64), 2.18-9.84 (2.25-7.13), and 2.76-6.51 (2.82-6.28) ppm, respectively, harvested at a 56-day PHI.

In the residue decline trials, residues of combined dicamba and 5-OH dicamba generally decreased with increasing PHI in/on forage and hay.

An acceptable method was used for residue quantitation, and adequate storage stability data are available to support sample storage durations and conditions for all analytes.

REFERENCES

48001303.DER, A. Kamel, 4/30/13
Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger

B.7.6 Residues Resulting from Supervised Trials (Annex IIA 6.3; Annex IIIA 8.3)

B.7.6.1 Residues in Target Crops

B.7.6.1.3. Wheat (Bridging data)

Document ID:	MRID No. 49379303
Report:	Shepard, E. (2013) Formulation Bridging Study Magnitude of the Residue of Dicamba in Wheat after Application of BAS 183 H, BAS 183 UYH, or BAS 183 WBH (Clarity Herbicide and Two New Salt Formulations). BASF Study Number 389563. BASF Reg. Doc. No. 2012/7005467. Unpublished study prepared by BASF Crop Protection. 323 p.
Guidelines:	EPA OCSPP Harmonized Test Guideline 860.1500 Crop Field Trials (August 1996) PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 9 – Crop Field Trials PMRA Regulatory Directive DIR2010-05 – Revisions to the Residue Chemistry Crop Field Trial Requirements OECD Guideline 509 Crop Field Trial (September 2009)
GLP Compliance:	No deviations from regulatory requirements were reported which would have an impact on the validity of the study.
Acceptability:	The study is considered scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document, DP# D429868.
Evaluator:	Peter Savoia, Chemist, Registration Action Branch V/VII/Health Effects Division

Note: This Data Evaluation Record (DER) was originally prepared under contract by Versar, Inc. (6850 Versar Center, Springfield, VA 22151; submitted 12/15/14. The DER has been reviewed by HED and revised to reflect current Office of Pesticide Programs (OPP) policies.

EXECUTIVE SUMMARY

BASF Crop Protection has submitted field trial data for dicamba on wheat. Four field trials were conducted in the United States during the 2010 growing season in the North American Free Trade Agreement (NAFTA) Growing Zones 5 (IA and MN, 2 trials), 7 (NE, 1 trial), and 11 (WA, 1 trial). The study was submitted to provide bridging data in support of two new salt formulations of dicamba, the 4 lb ae/gal (480 g ae/L) SL formulation containing diethylenetriamine (DETA) salt (BAS 183 UYH) and the 5 lb ae/gal (600 g ae/L) SL formulation containing N,N-bis-(3-aminopropyl)methylamine (BAPMA) salt (BAS 183 WBH), through side-by-side trials conducted with the 4 lb ae/gal (480 g ae/L) SL formulation containing 2-(2-aminoethoxy)ethanol (a.k.a. diglycolamine; DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137).

Each field site consisted of one untreated plot (Plot 1) and three side-by-side treated plots (Plots 2, 3, and 4) reflecting application of three soluble concentrate (SL) formulations of dicamba each containing a different amine salt: the 4 lb ae/gal (480 g ae/L) DGA SL formulation, the 4 lb ae/gal (480 g ae/L) DETA SL formulation, and the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation. Each treated plot received one preplant soil application at 0.123-0.132 lb acid equivalent (ae)/A (0.138-0.148 kg ae/ha) followed by two foliar broadcast applications at 0.241-0.262 lb ae/A/application (0.270-0.293 kg ae/ha/application). The second application was made at the six-leaf growth stage, 31-45 days after the preplant application. There was a 50- to 68-day retreatment interval (RTI) between the second and third foliar applications. Applications were made using ground equipment, in spray volumes of ~14-20 gal/A (128-188 L/ha) of water. A nonionic surfactant (NIS) was added to spray mixtures for each trial. Samples of wheat forage and hay were harvested at preharvest interval (PHI) of 7 days after the second application. Hay was allowed to dry in the field for 5-8 days after harvest. Samples of wheat grain and straw were harvested at a PHI of 6-7 days after the third application. Total application rates were 0.366-0.388 lb ae/A (0.410-0.434 kg ai/ha) for wheat forage and hay and 0.615-0.650 lb ae/A (0.689-0.724 kg ae/ha) for grain and straw.

All samples were maintained frozen at the testing facility, during shipping, and were stored frozen at the sample preparation facility and at the analytical laboratories until analysis. The field residue samples were stored frozen a maximum of 310 days (10.2 months) for forage, 307 days (10.1 months) for hay, 996 days (32.8 months) for grain, and 960 days (31.6 months) for straw from harvest to extraction. Samples were analyzed within 0-16 days of extraction. Adequate storage stability data are available to support the study reflecting the stability of residues of dicamba and 5-OH dicamba under frozen storage conditions in/on wheat forage and hay for up to 258 and 283 days (8.5 and 9.3 months), respectively, and field corn forage, silage, grain, and fodder for up to 3 and 2 years, respectively (refer to the Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger). Adequate storage stability data are therefore available to support the storage conditions and intervals for samples in the current trials.

Samples were analyzed for residues of dicamba and its metabolite 5-OH dicamba using high performance liquid chromatography with tandem mass spectrometric detection (LC/MS/MS); Method D0902 (and BASF REG DOC No. 97/5441) with minor modifications. The limit of quantitation (LOQ; determined as the lowest limit of method validation, LLMV) was 0.01 ppm for all analytes in all wheat matrices. The method was verified prior to and in conjunction with sample analysis and is considered adequate based on acceptable method validation and concurrent recovery data. The fortification levels used in concurrent method recovery were adequate to bracket expected residue levels. Concurrent recoveries were corrected for apparent residues in controls; treated samples were not corrected for residues in controls. Quantifiable residues of 5-OH dicamba were converted to parent equivalents by the registrant using a molecular weight conversion factor of 0.9325.

When the field trial results for the three formulations are compared, residues in/on all wheat matrices resulting from application of the 4 lb ae/gal (480 g ae/L) DGA SL formulation of dicamba were higher than those resulting from either the 4 lb ae/gal (480 g ae/L) DETA SL formulation or the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation.

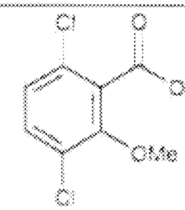
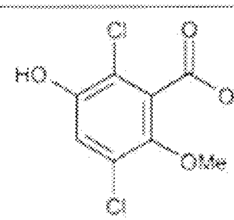
Following two applications (single preplant followed by a single broadcast foliar application) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 0.366-0.388 lb ae/A (0.410-0.434 kg ae/ha), combined residues (and per trial averages) of dicamba and 5-OH dicamba were 3.89-7.00 (3.96-6.57), 3.71-5.61 (4.32-5.41), and 3.10-5.73 (3.15-5.28) ppm, respectively, in/on **wheat forage** harvested at a PHI of 7 days. Corresponding residues in/on **wheat hay** were 8.15-17.2 (8.42-16.8), 6.13-14.5 (6.46-13.3), and 6.23-14.2 (6.56-13.7) ppm, respectively.

Following three applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 0.615-0.650 lb ae/A (0.689-0.727 kg ae/ha), combined residues (and per trial averages) of dicamba and 5-OH dicamba were <0.0919-1.76 (<0.0160-1.73), <0.0619-0.911 (<0.0890-0.863), and <0.0827-0.929 (<0.0979-0.905) ppm, respectively, in/on **wheat grain** harvested at a PHI of 6-7 days. Corresponding residues in/on **wheat straw** were 6.72-32.0 (7.65-31.7), 5.72-27.9 (6.04-27.7), and 3.71-24.6 (4.85-24.3) ppm, respectively.

No residue decline trials were conducted.

I. MATERIALS AND METHODS

A. MATERIALS

Table B.7.6.1.3-1. Nomenclature for Dicamba.	
Common name	Dicamba acid
Identity	3,6-dichloro-2-methoxybenzoic acid
CAS no.	1918-00-9 (dicamba acid) or 1982-69-0 (sodium salt of dicamba)
Company experimental name	BAS 183 H
Other synonyms (if applicable)	N/A
	
Common name	5-Hydroxy-dicamba
Identity	3,6-dichloro-5-hydroxy-2-methoxy-benzoic acid
CAS no.	7600-50-2
Company experimental name	5-OH dicamba
Other synonyms (if applicable)	N/A
	

B. Study Design

1. Test Procedure

A total of four side-by-side residue trials in/on wheat were conducted with three 4 or 5 lb ae/gal SL formulations during the 2010 growing season. Field trial locations by NAFTA growing zone are summarized in Table B.7.6.1.3.2.

Table B.7.6.1.3-2. Trial Numbers and Geographical Locations.

Crop	No. Trials	NAFTA Growing Zone												Total
		1	2	3	4	5	6	7	8	9	10	11	12	
Wheat	Sub.					2		1				1		4
	Req.[1]		1/1		1/1	5/3	1/1	5/4	6/4			1/1		20/15

[1] As per Table 5 of 860.1500 for wheat; the second number reflects a 25% reduction in the number of field trials allowed for the crop as a representative commodity in support of a crop group/subgroup tolerance or when application results in no quantifiable residues.

Locations and detailed use patterns for the trials are provided in Table B.7.6.1.3-3.

Table B.7.6.1.3-3. Study Use Pattern.

Location: City, State; Year (Trial ID)	Plot	End-use Product[1]	Method of Application/ Timing of Application	Volume (gal/A) [L/ha]	Rate per Application (lb ae/A) [kg ae/ha]	Retreatment Interval (days)	Total Rate[2] (lb ae/A) [kg ae/ha]	Surfactant/ Adjuvant[3]
Richland, IA; 2010 (RCN R100184)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	13.7 [128]	0.123 [0.138]	--	0.366 [0.410]	NIS
			2. Broadcast foliar spray; 6 leaf	15.7 [147]	0.243 [0.272]	34		NIS
			3. Broadcast foliar spray; BBCH 97	16.2 [152]	0.250 [0.280]	68	0.616 [0.690]	NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	13.7 [128]	0.124 [0.139]	--	0.375 [0.420]	NIS
			2. Broadcast foliar spray; 6 leaf	15.7 [147]	0.251 [0.281]	34		NIS
			3. Broadcast foliar spray; BBCH 97	16.2 [152]	0.253 [0.283]	68	0.628 [0.703]	NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	13.7 [128]	0.127 [0.142]	--	0.374 [0.419]	NIS
			2. Broadcast foliar spray; 6 leaf	15.7 [147]	0.247 [0.277]	34		NIS
			3. Broadcast foliar spray; BBCH 97	16.2 [152]	0.251 [0.281]	68	0.625 [0.700]	NIS
Geneva, MN; 2010 (RCN R100185)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	17.7 [166]	0.123 [0.138]	--	0.373 [0.418]	NIS
			2. Broadcast foliar spray; 6 leaf	17.2 [161]	0.250 [0.280]	31		NIS
			3. Broadcast foliar spray; mature	18.2 [170]	0.252 [0.282]	64	0.625 [0.700]	NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	17.7 [166]	0.124 [0.139]	--	0.372 [0.417]	NIS
			2. Broadcast foliar spray; 6 leaf	17.2 [161]	0.248 [0.278]	31		NIS

Table B.7.6.1.3-3. Study Use Pattern.

Location: City, State; Year (Trial ID)	Plot	End-use Product[1]	Method of Application/ Timing of Application	Volume (gal/A) [L/ha]	Rate per Application (lb ae/A) [kg ae/ha]	Retreatment Interval (days)	Total Rate[2] (lb ae/A) [kg ae/ha]	Surfactant/ Adjuvant[3]
			3. Broadcast foliar spray; mature	18.2 [170]	0.252 [0.282]	64	0.624 [0.699]	NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	17.7 [166]	0.124 [0.139]	--	0.372 [0.417]	NIS
			2. Broadcast foliar spray; 6 leaf	17.2 [161]	0.248 [0.278]	31		NIS
			3. Broadcast foliar spray; mature	18.2 [170]	0.253 [0.283]	64	0.625 [0.700]	NIS
Grand Island, NE; 2010 (RCN R100186)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	20.04 [187]	0.126 [0.141]	--	0.387 [0.433]	NIS
			2. Broadcast foliar spray; BBCH 30	20.08 [188]	0.261 [0.292]	41		NIS
			3. Broadcast foliar spray; BBCH 87	20.15 [188]	0.262 [0.293]	50	0.649 [0.726]	NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	20.04 [187]	0.126 [0.141]	--	0.388 [0.434]	NIS
			2. Broadcast foliar spray; BBCH 30	20.08 [188]	0.262 [0.293]	41		NIS
			3. Broadcast foliar spray; BBCH 87	20.15 [188]	0.262 [0.293]	50	0.650 [0.727]	NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	20.04 [187]	0.132 [0.148]	--	0.374 [0.419]	NIS
			2. Broadcast foliar spray; BBCH 30	20.08 [188]	0.242 [0.271]	41		NIS
			3. Broadcast foliar spray; BBCH 87	20.15 [188]	0.241 [0.270]	50	0.615 [0.689]	NIS
Ephrata, WA; 2010 (RCN R100187)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	20.0 [187]	0.125 [0.140]	--	0.375 [0.420]	NIS
			2. Broadcast foliar spray; BBCH 26	20.0 [187]	0.250 [0.280]	45		NIS
			3. Broadcast foliar spray; BBCH 87	20.0 [187]	0.252 [0.282]	66	0.627 [0.702]	NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	20.0 [187]	0.125 [0.140]	--	0.375 [0.420]	NIS
			2. Broadcast foliar spray; BBCH 26	20.0 [187]	0.250 [0.280]	45		NIS
			3. Broadcast foliar spray; BBCH 87	20.0 [187]	0.251 [0.281]	66	0.626 [0.701]	NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	20.0 [187]	0.127 [0.142]	--	0.378 [0.423]	NIS
			2. Broadcast foliar spray; BBCH 26	20.0 [187]	0.251 [0.281]	45		NIS
			3. Broadcast foliar spray; BBCH 87	20.0 [187]	0.252 [0.282]	66	0.630 [0.705]	NIS

1 EP = End-use product; Plot 2: 4 lb ae/gal (480 g ae/L) soluble concentrate (SL) formulation of dicamba as the 2-(2-aminoethoxy)ethanol (DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137); Plot 3: 4 lb ae/gal (480 g ae/L) SL formulation of dicamba as the diethylenetriamine (DETA) salt (DETA BAS 183 UYH); and Plot 4: 5 lb ae/gal (600 g ae/L) SL formulation as the N,N-Bis-(3-aminopropyl)methylamine (BPMA) salt (BPMA BAS 183 WBH).

2 Forage and hay were harvested after two applications.

3 NIS = nonionic surfactant.

Wheat was grown and maintained according to typical agricultural practices. Irrigation was used at the WA trial site. No unusual weather conditions were reported to have adversely affected crop production or yield during the study.

Sample Handling and Preparation

Single control and duplicate treated samples of wheat forage and hay were harvested by hand 7 days after the second application. Hay samples were allowed to dry in the field 5-8 days prior to collection. Single control and duplicate treated samples of wheat grain and straw were harvested by hand and threshed (IA and MN trials) or by a mechanized harvester (NE and WA trials) 6-7 days after the third application. The samples were commercially acceptable wheat RAC samples and consisted of at least 12 plants. RAC samples weighed ≥1 kg (forage and grain) and ≥0.5 kg (hay and straw). All treated samples were placed in frozen storage at the field sites within 3.5 hours of harvest/collection. All samples were shipped within 15 days of collection by freezer truck to BASF Agricultural Research Center (Research Triangle Park, NC) for homogenization, where they were maintained frozen (-20 °C) prior to preparation. In preparation for analysis, the samples were processed per SOP CHEM111.12 & CHEM111.13. Homogenized samples of all wheat matrices were shipped frozen on dry ice via FedEx to the analytical laboratory, Pyxant Labs., Inc. (Colorado Springs, CO). Wheat forage, hay, and grain samples were maintained frozen (<-20 °C) at Pyxant prior to analysis. Samples of wheat straw were subsequently shipped frozen via FedEx to the ADPEN Laboratories, Inc. (Jacksonville, FL) for residue analysis. At ADPEN, wheat straw samples were maintained frozen (-15 to -10 °C) prior to analysis.

2. Description of Analytical Procedures

Samples of wheat (forage, hay, grain, and straw) were analyzed for residues of dicamba and 5-OH dicamba using a modified version of LC/MS/MS method D0902. For analysis of wheat forage, hay, and grain minor modifications to instrumentation and conditions were made including: (i) decreasing column size and adjusting the gradient, flow rate, and MS conditions; (ii) adjusting column length as necessary to move interference from 5-OH dicamba; and (iii) adding a needle wash to reduce autosampler carryover. For analysis of wheat straw, minor modifications include: (i) reducing homogenized sample weight; (ii) adding a second extraction with 1 N HCl after initial filtration; and (iii) volume of methanol:water was reduced prior to LC/MS/MS. A brief description of the method was included in the submission. For a complete description and method validation refer to 48001303.DER, A. Kamel, 4/30/13.

Briefly, homogenized samples were heated with 1 N HCl at ~90 °C for ~45 minutes. The extract was cooled to room temperature and filtered, then adjusted to volume with water. The extract was adjusted to pH 9-10 with concentrated NH_4OH, vortexed, and adjusted to pH 3-4 with concentrated formic acid. Sodium chloride was added, and the extract was partitioned twice with hexane:ethyl acetate (1:1, v:v) followed by centrifugation. The resulting organic phase was reduced to dryness under nitrogen, then reconstituted in methanol:water (10:90, v:v) for analysis by LC/MS/MS. Two transition ions were monitored for each analyte.

The LOQ (determined as the LLMV), was 0.01 ppm for all analytes in the wheat matrices; the corresponding limit of detection (LOD) was 0.002 ppm (20% LOQ).

II. RESULTS AND DISCUSSION

Method performance was evaluated during method validation and by use of concurrent recovery samples. For method validation, samples of untreated wheat forage were fortified with each analyte at 7.0 ppm, samples of untreated wheat hay were fortified at 0.01, 0.1, and 17.0 ppm, and samples of untreated wheat grain were fortified at 2.99 ppm. For concurrent recovery, samples of wheat forage, hay, and grain were fortified with each analyte at 0.01 and 0.1 ppm and samples of wheat straw were fortified at 0.01, 0.1, 10, 40 ppm. Average recoveries were within the acceptable range of 70-120% with the exception of wheat hay. Average recoveries of dicamba and 5-OH dicamba in wheat hay were each 68% with a standard deviation of 9.4% for dicamba and 16% for 5-OH dicamba. The method was considered valid for the analysis of dicamba residues in/on wheat matrices (Table B.7.6.1.3-4). The fortification levels bracketed the measured residues. Concurrent recoveries were corrected for apparent residues in controls; residues in the treated samples were not corrected for apparent residues in controls.

For analysis of wheat forage, hay, and grain, the detector response was linear (coefficients of determination, $r^2 \geq 0.9869$ for dicamba and $r^2 \geq 0.9826$ for 5-OH dicamba within the range of 0.125-1.0 ng/mL). For analysis of wheat straw, the detector response was linear (coefficients of determination, $r^2 \geq 0.9986$ for dicamba and $r^2 \geq 0.9994$ for 5-OH dicamba within the range of 0.001-0.50 ng/mL). Representative chromatograms of control samples, fortified samples and treated samples were provided. The control chromatograms generally had no peaks of interest above the chromatographic background. The fortified sample chromatograms contained only the analyte of interest, and peaks were symmetrical and well defined. Apparent residues of each analyte in controls were <0.01 ppm with the following exceptions: apparent residues of dicamba were found in one untreated wheat straw sample (0.01 ppm) and residues of 5-OH dicamba were found in one untreated hay sample (0.0177 ppm) and three untreated wheat straw samples (0.01-0.02 ppm). The reported residue values were not corrected for apparent residues in controls. Residues of 5-OH dicamba are expressed in parent equivalents as calculated by the registrant using a molecular weight conversion factor of 0.9325.

Table B.7.6.1.3-4. Summary of Method Validation and Procedural/Concurrent Recoveries of Dicamba and 5-OH Dicamba from Wheat Matrices.

Matrix	Analyte	Fortification Level (ppm)	Sample size (n)	Recoveries[1] (%)	Mean ± Std. Dev. (%)
Method Validation Recoveries					
Wheat forage	Dicamba	7.0	3	74-82	74 ± 4.2
	5-OH Dicamba	7.0	3	81-88	84 ± 3.7
Wheat hay	Dicamba	0.01-17.0	12	60, 62, 67, 67, 67, 68, 69; 71-78	70 ± 5.9
	5-OH Dicamba	0.01-17.0	12	68; 74-99	84 ± 8.3
Wheat grain	Dicamba	2.99	3	71-77	74 ± 3.2
	5-OH Dicamba	2.99	3	69; 76, 78	74 ± 4.9
Concurrent Recoveries[2]					
Wheat forage	Dicamba	0.01-0.1	4	74-83	80 ± 4.0
	5-OH Dicamba	0.01-0.1	4	67; 82-90	82 ± 10
Wheat hay	Dicamba	0.01-0.1	4	62, 63, 64; 82	68 ± 9.4
	5-OH Dicamba	0.01-0.1	4	56, 61, 63; 91	68 ± 16
Wheat grain	Dicamba	0.01-0.1	6	60, 64, 69, 69; 78-85	71 ± 8.9

Table B.7.6.1.3-4. Summary of Method Validation and Procedural/Concurrent Recoveries of Dicamba and 5-OH Dicamba from Wheat Matrices.					
Matrix	Analyte	Fortification Level (ppm)	Sample size (n)	Recoveries[1] (%)	Mean ± Std. Dev. (%)
	5-OH Dicamba	0.01-0.1	6	61; 76-101	83 ± 14
Wheat straw	Dicamba	0.01-40	13	77-96	87 ± 5.8
	5-OH Dicamba	0.01-40	13	72-115	92 ± 13

[1] Concurrent recoveries were corrected for apparent residues in controls.
[2] Some values include mean values of duplicate injections of the same fortification sample.

The field residue samples were stored frozen a maximum of 310 days (10.2 months) for forage, 307 days (10.1 months) for hay, 996 days (32.8 months) for grain, and 960 days (31.6 months) for straw from harvest to extraction (Table B.7.6.1.3-5). Samples were analyzed within 0-16 days of extraction. Adequate storage stability data are available to support the study reflecting the stability of residues of dicamba and 5-OH dicamba under frozen storage conditions in/on wheat forage and hay for up to 258 and 283 days (8.5 and 9.3 months), respectively, and field corn forage, silage, grain, and fodder for up to 3 and 2 years, respectively (refer to the Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger). These data are acceptable to support the storage conditions and durations of samples from the submitted field trials.

Table B.7.6.1.3-5. Summary of Storage Conditions.			
Matrix	Storage Temperature (°C)	Actual Storage Duration[1]	Interval of Demonstrated Storage Stability
Wheat forage	Frozen at the field sites; ~-20 at BASF ~-20 at Pyxant -15 to -10 at ADPEN	286-310 days (9.4-10.2 months)	The available freezer storage stability data indicate that residues of dicamba and 5-OH dicamba are stable under frozen storage conditions in/on wheat forage and hay for up to 258 and 283 days (8.5 and 9.3 months), respectively, and field corn forage, silage, grain, and fodder for up to 3 and 2 years, respectively.[2]
Wheat hay		280-307 days (9.2-10.1 months)	
Wheat grain		225-996 days (7.4-32.8 months)	
Wheat straw		950-960 days (31.3-31.6 months)	

[1] Interval from harvest to extraction. Samples were analyzed within 0-16 days of extraction.
[2] Refer to the Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger

The results from the submitted field trials are presented in Tables B.7.6.1.3-6 and B.7.6.1.3-7. When the field trial results for the three formulations are compared, residues in/on all wheat matrices resulting from application of the 4 lb ae/gal (480 g ae/L) DGA SL formulation of dicamba were higher than those resulting from either the 4 lb ae/gal (480 g ae/L) DETA SL formulation or the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation.

Following two applications (single preplant followed by a single broadcast foliar application) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 0.366-0.388 lb ae/A (0.410-0.434 kg ae/ha), combined residues (and per trial averages) of dicamba and 5-OH dicamba were 3.89-7.00 (3.96-6.57), 3.71-5.61 (4.32-5.41), and 3.10-5.73 (3.15-5.28) ppm, respectively, in/on **wheat forage** harvested at a PHI of 7 days. Corresponding residues in/on **wheat hay** were 8.15-17.2 (8.42-16.8), 6.13-14.5 (6.46-13.3), and 6.23-14.2 (6.56-13.7) ppm, respectively.

Following three side-by-side applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 0.615-0.650 lb ae/A (0.689-0.727 kg ae/ha), combined residues (and per trial averages) of dicamba and 5-OH dicamba were <0.0919-1.76 (<0.0160-1.73), <0.0619-0.911 (<0.0890-0.863), and <0.0827-0.929 (<0.0979-0.905) ppm, respectively, in/on **wheat grain** harvested at a PHI of 6-7 days. Corresponding residues in/on **wheat straw** were 6.72-32.0 (7.65-31.7), 5.72-27.9 (6.04-27.7), and 3.71-24.6 (4.85-24.3) ppm, respectively.

No residue decline trials were conducted.

Table B.7.6.1.3-6. Residue Data from Wheat Field Trials with Dicamba.[1]

Location: City, State; Year (Trial ID)	Zone	Wheat/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
Richland, IA; 2010 (RCN R100184)	5	Briggs	2	Forage	0.366 [0.410]	7	1.10, 0.944 [1.02]	3.33, 2.95 [3.14]	4.43, 3.89 [4.16]
				Hay		7 (6)	1.90, 1.71 [1.81]	11.1, 10.9 [11.0]	13.0, 12.6 [12.8]
				Grain	0.616 [0.690]	7	1.66, 1.73 [1.70]	0.0350, 0.0269 [0.0310]	1.69, 1.76 [1.73]
				Straw		7	8.49, 6.68 [7.59]	0.0850, 0.0425 [0.0638]	8.57, 6.72 [7.65]
			3	Forage	0.375 [0.420]	7	0.955, 1.24 [1.09]	2.75, 3.69 [3.22]	3.71, 4.92 [4.32]
				Hay		7 (6)	2.83, 1.95 [2.39]	11.7, 9.3 [10.5]	14.5, 11.2 [12.9]
				Grain	0.628 [0.703]	7	0.896, 0.799 [0.848]	0.0147, 0.0145 [0.0146]	0.911, 0.814 [0.863]
				Straw		7	6.26, 5.69 [5.98]	0.0793, 0.0437 [0.0615]	6.34, 5.73 [6.04]
			4	Forage	0.374 [0.419]	7	0.843, 0.845 [0.844]	2.26, 2.36 [2.31]	3.10, 3.20 [3.15]
				Hay		7 (6)	1.73, 1.58 [1.66]	8.36, 7.90 [8.13]	10.1, 9.48 [9.79]
				Grain	0.625 [0.700]	7	0.867, 0.919 [0.893]	0.0132, <0.01 [<0.0116]	0.880, <0.929 [<0.905]
				Straw		7	5.94, 3.66 [4.80]	0.0439, 0.0520 [0.0480]	5.98, 3.71 [4.85]
Geneva, MN; 2010 (RCN R100185)	5	Steele	2	Forage	0.373 [0.418]	7	1.10, 1.24 [1.17]	5.04, 5.76 [5.40]	6.14, 7.00 [6.57]
				Hay		7 (5)	1.01, 0.886 [0.948]	7.68, 7.26 [7.47]	8.69, 8.15 [8.42]
				Grain	0.625 [0.700]	6	0.159, 0.154 [0.157]	(0.00208), (0.00225) [<0.01]	<0.169, <0.164 [<0.167]
				Straw		6	13.6, 9.09 [11.3]	0.288, 0.300 [0.294]	13.9, 9.39 [11.6]

Table B.7.6.1.3-6. Residue Data from Wheat Field Trials with Dicamba.[1]

Location: City, State; Year (Trial ID)	Zone	Wheat/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
			3	Forage	0.372 [0.417]	7	1.18, 1.15 [1.17]	4.43, 4.05 [4.24]	5.61, 5.20 [5.41]
				Hay		7 (5)	0.941, 0.846 [0.894]	5.84, 5.29 [5.57]	6.78, 6.13 [6.46]
				Grain	0.624 [0.699]	6	0.172, 0.177 [0.175]	(0.00489), (0.00586) [<0.01]	<0.182, <0.187 [<0.185]
				Straw		6	9.96, 12.5 [11.2]	0.230, 0.259 [0.245]	10.2, 12.8 [11.5]
			4	Forage	0.372 [0.417]	7	0.915, 1.09 [1.00]	3.91, 4.64 [4.28]	4.82, 5.73 [5.28]
				Hay		7 (5)	0.893, 0.839 [0.866]	6.00, 5.39 [5.70]	6.89, 6.23 [6.56]
				Grain	0.625 [0.700]	6	0.137, 0.0776 [0.107]	(0.00612), (0.00295) [<0.01]	<0.147, <0.0876 [<0.117]
				Straw		6	10.5, 12.7 [11.6]	0.205, 0.269 [0.237]	10.7, 12.9 [11.8]
Grand Island, NE; 2010 (RCN R100186)	7	Traverse HRS	2	Forage	0.387 [0.433]	7	0.736, 0.710 [0.723]	4.95, 5.04 [5.00]	5.69, 5.75 [5.72]
				Hay		7 (7)	1.51, 1.65 [1.58]	14.8, 15.6 [15.2]	16.3, 17.2 [16.8]
				Grain	0.649 [0.726]	7	0.530, 0.548 [0.539]	0.0773[6], 0.0846[6] [0.0810]	0.607[6], 0.633[6] [0.620]
				Straw		7	7.13, 9.14 [8.14]	1.65, 2.08 [1.87]	8.78, 11.2 [10.0]
			3	Forage	0.388 [0.434]	7	0.692, 0.604 [0.648]	4.30, 4.21 [4.26]	4.99, 4.82 [4.91]
				Hay		7 (7)	1.37, 1.42 [1.40]	11.5, 12.3 [11.9]	12.8, 13.7 [13.3]
				Grain	0.650 [0.727]	7	0.310, 0.266 [0.288]	0.0155, 0.0131 [0.0143]	0.325, 0.279 [0.302]
				Straw		7	5.14, 6.74 [5.94]	0.579, 0.787 [0.683]	5.72, 7.53 [6.62]
			4	Forage	0.374 [0.419]	7	0.646, 0.664 [0.655]	4.35, 4.19 [4.27]	5.00, 4.85 [4.93]
				Hay		7 (7)	1.10, 1.29 [1.20]	9.79, 11.4 [10.6]	10.9, 12.7 [11.8]
				Grain	0.615 [0.689]	7	0.294, 0.117 [0.206]	0.0145 (0.00987) [<0.0123]	0.309, <0.127 [<0.218]
				Straw		7	7.75, 7.42 [7.59]	1.13, 0.888 [1.01]	8.88, 8.31 [8.60]

Table B.7.6.1.3-6. Residue Data from Wheat Field Trials with Dicamba.[1]

Location: City, State; Year (Trial ID)	Zone	Wheat/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
Ephrata, WA; 2010 (RCN R100187)	11	Alpowa	2	Forage	0.375 [0.420]	7	0.982, 0.972 [0.977]	2.99, 2.97 [2.98]	3.98, 3.94 [3.96]
				Hay		7 (8)	2.48, 2.77 [2.63]	10.2, 11.0 [10.6]	12.6, 13.8 [13.2]
				Grain	0.627 [0.702]	6	0.0716, 0.207 [0.139]	0.0203, 0.0218 [0.0211]	0.0919, 0.229 [0.160]
				Straw		6	30.1, 29.8 [29.9]	1.88, 1.63 [1.75]	32.0, 31.4 [31.7]
			3	Forage	0.375 [0.420]	7	1.14, 1.16 [1.15]	3.31, 3.32 [3.32]	4.45, 4.48 [4.47]
				Hay		7 (8)	2.56, 2.77 [2.67]	9.51, 9.88 [9.70]	12.1, 12.7 [12.4]
				Grain	0.626 [0.701]	6	0.105, 0.0519 [0.0785]	0.0115, (0.00563) [<0.0108]	0.116, <0.0619 [<0.0890]
				Straw		6	27.3, 27.1 [27.2]	0.595, 0.503 [0.549]	27.9, 27.6 [27.7]
			4	Forage	0.378 [0.423]	7	1.00, 1.06 [1.03]	3.03, 3.25 [3.14]	4.03, 4.31 [4.17]
				Hay		7 (8)	2.89, 2.84 [2.87]	10.4, 11.4 [10.9]	13.2, 14.2 [13.7]
				Grain	0.630 [0.705]	6	0.0727, 0.103 [0.0879]	(0.00720), 0.0104 [<0.0102]	<0.0827, 0.113 [<0.0979]
				Straw		6	23.2, 23.7 [23.4]	0.761, 0.980 [0.870]	23.9, 24.6 [24.3]

[1] End-use product: Plot 2: 4 lb ae/gal (480 g ae/L) soluble concentrate (SL) formulation of dicamba as the 2-(2-aminoethoxy)ethanol (DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137); Plot 3: 4 lb ae/gal (480 g ae/L) SL formulation of dicamba as the diethylenetriamine (DETA) salt (DETA BAS 183 UYH); and Plot 4: 5 lb ae/gal (600 g ae/L) SL formulation as the N,N-Bis-(3-aminopropyl)methylamine (BPMA) salt (BPMA BAS 183 WBH).
[2] The number of days hay was allowed to dry in the field is reported in parentheses.
[3] The LOD was 0.002 ppm, and the LOQ was 0.01 ppm for each analyte. Values between the LOD and LOQ are reported in parenthesis. Residues below the LOQ were not converted to parent equivalents. For samples with nonquantifiable residues, combined residues and per trial averages were calculated by the study reviewer using the LOQ for residues <LOQ. For samples with quantifiable residues, combined residues and per trial averages were calculated by the registrant.
[4] Residues of 5-OH dicamba are expressed in parent equivalents as calculated by the registrant using a molecular weight conversion factor of 0.9325.
[5] Combined residues are the sum of dicamba and 5-OH dicamba.
[6] Mean value of duplicate injections of a single sample.

Table B.7.6.1.3-7. Summary of Residues from Wheat Field Trials with Dicamba.

Crop Matrix	Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
					Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Wheat forage	4 lb ae/gal DGA SL	0.366-0.387 [0.410-0.433]	7	4	3.89	7.00	3.96	6.57	4.94	5.10	1.26
	4 lb ae/gal DETA SL	0.372-0.388 [0.417-0.434]	7	4	3.71	5.61	4.32	5.41	4.69	4.77	0.490
	5 lb ae/gal BAPMA SL	0.372-0.378 [0.417-0.423]	7	4	3.10	5.73	3.15	5.28	4.55	4.45	0.941

Table B.7.6.1.3-7. Summary of Residues from Wheat Field Trials with Dicamba.											
Crop Matrix	Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
					Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Wheat hay	4 lb ae/gal DGA SL	0.366-0.387 [0.410-0.433]	7	4	8.15	17.2	8.42	16.8	13.0	12.8	3.41
	4 lb ae/gal DETA SL	0.372-0.388 [0.417-0.434]	7	4	6.13	14.5	6.46	13.3	12.6	11.2	3.21
	5 lb ae/gal BAPMA SL	0.372-0.378 [0.417-0.423]	7	4	6.23	14.2	6.56	13.7	10.8	10.5	3.05
Wheat grain	4 lb ae/gal DGA SL	0.616-0.649 [0.690-0.726]	6-7	4	<0.0919	1.76	0.0160	1.73	0.393	0.668	0.737
	4 lb ae/gal DETA SL	0.624-0.650 [0.699-0.727]	6-7	4	<0.0619	0.911	<0.0890	0.863	0.243	0.359	0.346
	5 lb ae/gal BAPMA SL	0.615-0.630 [0.689-0.705]	6-7	4	<0.0827	0.929	<0.0979	0.905	0.167	0.334	0.384
Wheat straw	4 lb ae/gal DGA SL	0.616-0.649 [0.690-0.726]	6-7	4	6.72	32.0	7.65	31.7	10.8	15.2	11.1
	4 lb ae/gal DETA SL	0.624-0.650 [0.699-0.727]	6-7	4	5.72	27.9	6.04	27.7	9.05	13.0	10.1
	5 lb ae/gal BAPMA SL	0.615-0.630 [0.689-0.705]	6-7	4	3.71	24.6	4.85	24.3	10.2	12.4	8.4

[1] n = number of field trials.
[2] Values based on total number of samples.
[3] Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation. For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (0.01 ppm).

III. CONCLUSIONS

The wheat field trials are considered scientifically acceptable. When the field trial results for the three formulations are compared, residues in/on all wheat matrices resulting from application of the 4 lb ae/gal (480 g ae/L) DGA SL formulation of dicamba were higher than those resulting from either the 4 lb ae/gal (480 g ae/L) DETA SL formulation or the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation.

Following two applications (single preplant followed by a single broadcast foliar application) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 0.366-0.388 lb ae/A (0.410-0.434 kg ae/ha), combined residues (and per trial averages) of dicamba and 5-OH dicamba were 3.89-7.00 (3.96-6.57), 3.71-5.61 (4.32-5.41), and 3.10-5.73 (3.15-5.28) ppm, respectively, in/on **wheat forage** harvested at a PHI of 7 days. Corresponding residues in/on **wheat hay** were 8.15-17.2 (8.42-16.8), 6.13-14.5 (6.46-13.3), and 6.23-14.2 (6.56-13.7) ppm, respectively.

Following three side-by-side applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 0.615-0.650 lb ae/A (0.689-0.727 kg ae/ha), combined residues (and per trial averages) of dicamba and 5-OH dicamba were <0.0919-1.76 (<0.0160-1.73), <0.0619-0.911 (<0.0890-0.863), and <0.0827-0.929 (<0.0979-0.905) ppm, respectively, in/on **wheat grain** harvested at a PHI of 6-7 days. Corresponding residues in/on wheat straw were 6.72-32.0 (7.65-31.7), 5.72-27.9 (6.04-27.7), and 3.71-24.6 (4.85-24.3) ppm, respectively.

No residue decline trials were conducted.

An acceptable method was used for residue quantitation, and adequate storage stability data are available to support sample storage durations and conditions for all analytes.

REFERENCES

48001303.DER, A. Kamel, 4/30/13
Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger

B.7.6 Residues Resulting from Supervised Trials (Annex IIA 6.3; Annex IIIA 8.3)

B.7.6.1 Residues in Target Crops

B.7.6.1.2. Field corn (Bridging data)

Document ID:	MRID No. 49379304
Report:	Shepard, E. (2013) Formulation Bridging Study: Magnitude of the Residue of Dicamba in Corn after Application of BAS 183 09H, BAS 183 UYH, or BAS 183 WBH (Clarity Herbicide and Two New Salt Formulations). BASF Study Number 389564. BASF Reg. Doc. No. 2012/7005468. Unpublished study prepared by BASF Crop Protection. 176 p.
Guidelines:	EPA OCSPP Harmonized Test Guideline 860.1500 Crop Field Trials (August 1996) PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 9 – Crop Field Trials PMRA Regulatory Directive DIR2010-05 – Revisions to the Residue Chemistry Crop Field Trial Requirements OECD Guideline 509 Crop Field Trial (September 2009)
GLP Compliance:	No deviations from regulatory requirements were reported which would have an impact on the validity of the study.
Acceptability:	The study is considered scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document, DP# D429868.
Evaluator:	Peter Savoia, Chemist, Registration Action Branch V/VII/Health Effects Division

Note: This Data Evaluation Record (DER) was originally prepared under contract by Versar, Inc. (6850 Versar Center, Springfield, VA 22151; submitted 12/15/14. The DER has been reviewed by HED and revised to reflect current Office of Pesticide Programs (OPP) policies.

EXECUTIVE SUMMARY

BASF Crop Protection has submitted field trial data for dicamba on field corn. Four field trials were conducted in the United States during the 2010 growing season in the North American Free Trade Agreement (NAFTA) Growing Zone 5 (IA, MN, and NE). The study was submitted to provide bridging data in support of two new salt formulations of dicamba, the 4 lb ae/gal (480 g ae/L) SL formulation containing diethylenetriamine (DETA) salt (BAS 183 UYH) and the 5 lb ae/gal (600 g ae/L) SL formulation containing N,N-bis-(3-aminopropyl)methylamine (BAPMA) salt (BAS 183 WBH), through side-by-side trials conducted with the 4 lb ae/gal (480 g ae/L) SL formulation containing 2-(2-aminoethoxy)ethanol (a.k.a. diglycolamine; DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137).

Each field site consisted of one untreated plot (Plot 1) and three and three side-by-side treated plots (Plots 2, 3, and 4) reflecting application of three soluble concentrate (SL) formulations of

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dicamba each containing a different amine salt: the 4 lb ae/gal (480 g ae/L) DGA SL formulation, the 4 lb ae/gal (480 g ae/L) DETA SL formulation, and the 5 lb ae/gal (600 g ae/L) BAPMA SL formulation. Each treated plot received one preplant soil application at 0.489-0.515 lb acid equivalent (ae)/A (0.548-0.577 kg ae/ha) followed by two foliar broadcast applications made at 0.482-0.508 lb ae/A (0.540-0.569 kg ae/ha) and 0.242-0.262 lb ae/A (0.271-0.293 kg ae/ha), respectively, for total application rate of 1.22-1.28 lb ae/A (1.37-1.43 kg ae/ha). The second application was made at the V3 (BBCH 13) growth stage 17-23 days after the preplant application. The third application was made at the V7-V9 (BBCH 18) growth stage with an 18- to 34-day retreatment interval (RTI) between the second and third foliar applications. Applications were made using ground equipment, in spray volumes of ~15-20 gal/A (139-190 L/ha) of water. A nonionic surfactant (NIS) was added to spray mixtures for each trial. Samples of corn forage were harvested at a preharvest interval (PHI) of 43-52 days and samples of corn grain and stover were harvested at a PHI of 90-99 days after the last foliar application.

All samples were maintained frozen at the testing facility, during shipping to the laboratory, and were stored frozen until analysis. The field residue samples were stored frozen a maximum of 574 days (18.9 months) for forage, 538 days (17.7 months) for grain, and 541 days (17.8 months) for stover from harvest to extraction. Samples were analyzed on the same day of extraction. Adequate storage stability data are available indicating that residues of dicamba and 5-OH dicamba are stable under frozen storage conditions for up to 3 and 2 years, respectively, in/on field corn forage, silage, grain, and fodder (Dicamba RED, DP#317699, 12/20/05, C. Olinger). Adequate storage stability data are therefore available to support the storage conditions and intervals for samples in the current trials.

Samples were analyzed for residues of dicamba and its metabolite 5-OH dicamba using high pressure liquid chromatography with tandem mass spectrometric detection (LC/MS/MS); PSL RA006, which is based upon BASF Method D0902 with minor modifications. The limit of quantitation (LOQ; determined as the lowest limit of method validation, LLMV) was 0.01 ppm for all analytes in all corn matrices. The method was verified in conjunction with sample analysis and is considered adequate based on acceptable concurrent recovery data. The fortification levels used in concurrent method recovery were adequate to bracket expected residue levels. Concurrent recoveries were not corrected for apparent residues in controls; treated samples were not corrected for residues in controls. Quantifiable residues of 5-OH dicamba were converted to parent equivalents by the registrant using molecular weight conversion factor of 0.9325.

The results of the side-by-side trials suggest that combined residues of dicamba and 5-OH dicamba resulting from application of the three SL formulations prepared as different amine salts are comparable in corn matrices. Residues following application of the DETA salt and BAPMA salt are similar, but the highest residues occurred resulted from application of the DETA salt in/on corn forage and stover. Residues resulting from both the DETA and BAPMA salts are higher than the DGA salt. Combined residues resulting from application of the DGA, DETA, and BAPMA salts were at or below the LOQ in/on all samples of corn grain.

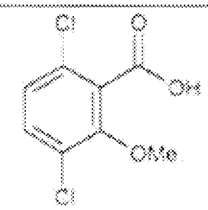
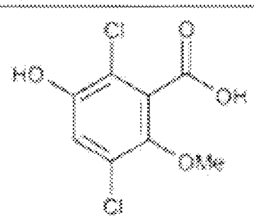
Following three applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 1.22-1.28 lb ae/A (1.37-1.43 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba were <0.02-0.667 (<0.239-<0.504), <0.0711-0.777 (<0.0836-0.707), and <0.0471-0.762 (<0.0594-0.623) ppm, respectively, in/on **corn forage** harvested at a PHI of 43-52 days.

Following three applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 1.22-1.28 lb ae/A (1.37-1.43 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba were <0.02-<0.0234 (<0.02-<0.0228), below the LOQ (<0.02), and <0.02-<0.0206 (<0.02-<0.0203) ppm, respectively, in/on **corn grain** harvested at a PHI of 90-99 days. Corresponding residues in/on **corn stover** were <0.0737-<0.453 (<0.0881-<0.430), <0.0306-0.680 (<0.0318-0.675), and <0.02-0.667 (<0.02-0.616) ppm.

No residue decline trials were conducted.

I. MATERIALS AND METHODS

A. MATERIALS

Table B.7.6.1.2-1. Nomenclature for Dicamba.	
Common name	Dicamba
Identity	3,6-dichloro-2-methoxybenzoic acid
CAS no.	1918-00-9 (dicamba acid) or 1982-69-0 (sodium salt of dicamba)
Company experimental name	BAS 183 H
Other synonyms (if applicable)	N/A
	
Common name	5-Hydroxy-dicamba
Identity	3,6-dichloro-5-hydroxy-2-methoxy-benzoic acid
CAS no.	7600-50-2
Company experimental name	5-OH dicamba
Other synonyms (if applicable)	N/A
	

B. Study Design

1. Test Procedure

A total of four side-by-side residue trials in/on corn were conducted with three 4 or 5 lb ae/gal SL formulations during the 2010 growing season. Field trial locations by NAFTA growing zone are summarized in Table B.7.6.1.2.2.

Table B.7.6.1.2-2. Trial Numbers and Geographical Locations.

Crop	No. Trials	NAFTA Growing Zone												Total
		1	2	3	4	5	6	7	8	9	10	11	12	
Corn	Sub.					4								4
	Req.[1]	1/1	1/1			17/12	1/1							20/15

[1] As per Table 5 of 860.1500 for corn; the second number reflects a 25% reduction in the number of field trials allowed for the crop as a representative commodity in support of a crop group/subgroup tolerance or when application results in no quantifiable residues.

Locations and detailed use patterns for the trials are provided in Table B.7.6.1.2-3.

Table B.7.6.1.2-3. Study Use Pattern.

Location: City, State; Year (Trial ID)	Plot	End-use Product[1]	Method of Application/ Timing of Application	Volume (gal/A) [L/ha]	Rate per Application (lb ae/A) [kg ae/ha]	Retreat-ment Interval (days)	Total Rate (lb ae/A) [kg ae/ha]	Surfactant / Adjuvant[2]
Jefferson, IA; 2010 (R100231)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	19.5 [182]	0.497 [0.557]	--	1.24 [1.39]	NIS
			2. Broadcast foliar spray; V3/BBCH 13	15.8 [148]	0.496 [0.556]	22		NIS
			3. Broadcast foliar spray; V8/BBCH 18	14.9 [139]	0.249 [0.279]	20		NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	19.5 [182]	0.490 [0.549]	--	1.22 [1.37]	NIS
			2. Broadcast foliar spray; V3/BBCH 13	15.8 [148]	0.482 [0.540]	22		NIS
			3. Broadcast foliar spray; V8/BBCH 18	14.9 [139]	0.249 [0.279]	20		NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	19.5 [182]	0.489 [0.548]	--	1.23 [1.37]	NIS
			2. Broadcast foliar spray; V3/BBCH 13	15.8 [148]	0.495 [0.554]	22		NIS
			3. Broadcast foliar spray; V8/BBCH 18	14.9 [139]	0.243 [0.272]	20		NIS
Freeborn, MN; 2010 (R100232)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	16.95 [159]	0.512 [0.573]	--	1.27 [1.42]	NIS
			2. Broadcast foliar spray; V3	17.4 [163]	0.508 [0.569]	23		NIS
			3. Broadcast foliar spray; V7-V9	17.7 [166]	0.252 [0.282]	31		NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	16.95 [159]	0.515 [0.577]	--	1.27 [1.42]	NIS
			2. Broadcast foliar spray; V3	17.4 [163]	0.505 [0.566]	23		NIS

Table B.7.6.1.2-3. Study Use Pattern.

Location: City, State; Year (Trial ID)	Plot	End-use Product[1]	Method of Application/ Timing of Application	Volume (gal/A) [L/ha]	Rate per Application (lb ae/A) [kg ae/ha]	Retreatment Interval (days)	Total Rate (lb ae/A) [kg ae/ha]	Surfactant / Adjuvant[2]
			3. Broadcast foliar spray; V7-V9	17.7 [166]	0.249 [0.278]	31		NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	16.95 [159]	0.510 [0.571]	--	1.26 [1.42]	NIS
			2. Broadcast foliar spray; V3	17.4 [163]	0.502 [0.562]	23		NIS
			3. Broadcast foliar spray; V7-V9	17.7 [166]	0.252 [0.282]	31		NIS
York, NE; 2010 (R100233)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	19.55 [183]	0.490 [0.549]	--	1.25 [1.39]	NIS
			2. Broadcast foliar spray; V3	19.87 [186]	0.497 [0.557]	17		NIS
			3. Broadcast foliar spray; V8	20.26 [190]	0.258 [0.289]	18		NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	19.55 [183]	0.490 [0.549]	--	1.24 [1.39]	NIS
			2. Broadcast foliar spray; V3	19.87 [186]	0.495 [0.554]	17		NIS
			3. Broadcast foliar spray; V8	20.26 [190]	0.258 [0.289]	18		NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	19.55 [183]	0.490 [0.549]	--	1.24 [1.39]	NIS
			2. Broadcast foliar spray; V3	19.87 [186]	0.499 [0.559]	17		NIS
			3. Broadcast foliar spray; V8	20.26 [190]	0.248 [0.278]	18		NIS
Polk, NE; 2010 (R100234)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	19.98 [187]	0.510 [0.571]	--	1.28 [1.43]	NIS
			2. Broadcast foliar spray; V3	20.11 [188]	0.504 [0.564]	20		NIS
			3. Broadcast foliar spray; V7-V8	20.11 [188]	0.261 [0.292]	34		NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	19.98 [187]	0.510 [0.571]	--	1.28 [1.43]	NIS
			2. Broadcast foliar spray; V3	20.11 [188]	0.504 [0.564]	20		NIS
			3. Broadcast foliar spray; V7-V8	20.11 [188]	0.262 [0.293]	34		NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	19.98 [187]	0.51 [0.571]	--	1.26 [1.41]	NIS
			2. Broadcast foliar spray; V3	20.11 [188]	0.504 [0.564]	20		NIS
			3. Broadcast foliar spray; V7-V8	20.11 [188]	0.242 [0.271]	34		NIS

[1] EP = End-use product; Plot 2: 4 lb ae/gal (480 g ae/L) soluble concentrate (SL) formulation of dicamba as the 2-(2-aminoethoxy)ethanol (DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137); Plot 3: 4 lb ae/gal (480 g ae/L) SL formulation of dicamba as the diethylenetriamine (DETA) salt (DETA BAS 183 UYH); and Plot 4: 5 lb ae/gal (600 g ae/L) SL formulation as the N,N-Bis-(3-aminopropyl)methylamine (BPMA) salt (BPMA BAS 183 WBH).
[2] NIS = nonionic surfactant.

Corn was grown and maintained according to typical agricultural practices. Irrigation was used at the NE trial sites. No unusual weather conditions were reported to have adversely affected crop production or yield during the study.

Sample Handling and Preparation

Single control and duplicate treated samples of corn forage were harvested by hand 43-52 days after the last foliar application. Single control and duplicate treated samples of corn grain and stover were harvested by hand 90-99 days after the last foliar application. The samples were commercially acceptable corn RAC samples and consisted of at least 12 plants. Each RAC sample weighed ≥1 kg (forage and grain) and ≥0.5 kg (stover). All treated samples were placed in frozen storage at the field trials within 3.5 hours of harvest. All samples were shipped within 23 days of collection by ACDS freezer truck to BASF Agricultural Research Center (Research Triangle Park, NC) for homogenization, where they were maintained frozen (temperature unspecified) prior to preparation. In preparation for analysis, the samples were processed per SOP CHEM111.12 & CHEM111.13. Homogenized samples of corn forage, grain, and stover were shipped frozen to the analytical laboratory, Product Safety Laboratories (PSL; Dayton, NJ) for residue analysis. Corn forage, grain, and stover samples were maintained frozen (<-18 °C) at the analytical laboratory prior to analysis.

2. Description of Analytical Procedures

Samples of corn (forage, grain, and stover) were analyzed for residues of dicamba and 5-OH dicamba using an LC/MS/MS modified version of method PSL RA006, developed by PSL, which is based on BASF Method D0902. A brief description of the method was included in the submission.

Briefly, homogenized samples were heated with 1 N HCl at ~90 °C for ~45 minutes. The extract was cooled to room temperature and filtered. The extract was partitioned (3x) with hexane:ethyl acetate (1:1, v:v) followed by centrifugation. The resulting organic phases were combined, concentrated NH_4OH was added, and reduced to near dryness under nitrogen. Residues were then reconstituted in methanol:water (1:9, v:v) for analysis by LC/MS/MS. Two transition ions were monitored for each analyte.

The LOQ (determined as the LLMV), was 0.01 ppm for all analytes in the corn matrices; the corresponding limit of detection (LOD) was 0.002 ppm.

II. RESULTS AND DISCUSSION

Method performance was evaluated by use of concurrent recovery samples. Samples of corn forage, grain, and stover were fortified with each analyte at 0.01 and 0.4 ppm. Recoveries were generally within the acceptable range of 70-120%. The method was considered valid for the analysis of dicamba residues in/on corn matrices (Table B.7.6.1.2-4). The fortification levels bracketed the measured residues. Concurrent recoveries were not corrected for apparent residues in controls; residues in the treated samples were not corrected for apparent residues in controls.

For analysis of corn forage, grain, and stover, the detector response was linear (coefficient of determination, $r^2 \geq 0.9976$ for dicamba and $r^2 \geq 0.9948$ for 5-OH dicamba within the range of 0.35-35 ng/mL). Representative chromatograms of control samples, fortified samples, and treated samples were provided. The control chromatograms generally had no peaks of interest above the chromatographic background. The fortified sample chromatograms contained only the analyte of interest, and peaks were symmetrical and well defined. Apparent residues of each analyte in controls were <0.01 ppm. The reported residue values were not corrected for apparent residues in controls. Residues of 5-OH dicamba are expressed in parent equivalents as calculated by the registrant using a molecular weight conversion factor of 0.9325.

Table B.7.6.1.2-4. Summary of Procedural/Concurrent Recoveries of Dicamba and 5-OH Dicamba from Corn Matrices.

Matrix	Analyte	Fortification Level (ppm)	Sample size (n)	Recoveries[1] (%)	Mean ± Std. Dev. (%)
Corn, forage	Dicamba	0.01-0.40	10	91-106	98 ± 5.3
	5-OH Dicamba	0.01-0.40	10	91-111; 140	103 ± 14.2
Corn, grain	Dicamba	0.01-0.40	8	76-97	84 ± 8.0
	5-OH Dicamba	0.01-0.40	8	76-107	89 ± 11.1
Corn, stover	Dicamba	0.01-0.40	8	75-96	84 ± 6.1
	5-OH Dicamba	0.01-0.40	8	75-94	82 ± 5.8

[1] Concurrent recoveries were not corrected for apparent residues in controls.

The field residue samples were stored frozen a maximum of 574 days (18.9 months) for forage, 538 days (17.7 months) for grain, and 541 days (17.8 months) for stover from harvest to extraction (Table B.7.6.1.2-5). Samples were analyzed the same day of extraction. Adequate storage stability data are available indicating that residues of dicamba and 5-OH dicamba are stable under frozen storage conditions for up to 3 and 2 years, respectively, in/on field corn forage, silage, grain, and fodder (Dicamba RED, DP#317699, 12/20/05, C. Olinger). These data are acceptable to support the storage conditions and durations of samples from the submitted field trials.

Table B.7.6.1.2-5. Summary of Storage Conditions.

Matrix	Storage Temperature (°C)	Actual Storage Duration[1]	Interval of Demonstrated Storage Stability
Corn, forage	<-18	564-574 days (18.6-18.9 months)	Adequate storage stability data are available indicating that residues of dicamba and 5-OH dicamba are stable under frozen storage conditions for up to 3 and 2 years, respectively, in/on field corn forage, silage, grain, and fodder.[2]
Corn, grain		528-538 days (17.4-17.7 months)	
Corn, stover		531-541 days (17.5-17.8 months)	

[1] Interval from harvest to extraction. Samples were analyzed the same day of extraction.
[2] Dicamba RED, DP#317699, 12/20/05, C. Olinger.

The results from the submitted field trials are presented in Tables B.7.6.1.2-6 and B.7.6.1.2-7. The results of the side-by-side trials suggest that combined residues of dicamba and 5-OH dicamba resulting from application of the three SL formulations prepared as different amine salts are comparable in corn matrices. Residues following application of the DETA salt and BAPMA salt are similar, but the highest residues occurred resulted from application of the DETA salt in/on corn forage and stover. Residues resulting from both the DETA and BAPMA salts are higher than the DGA salt. Combined residues resulting from application of the DGA, DETA, and BAPMA salts were at or below the LOQ in/on all samples of corn grain.

Following three applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 1.22-1.28 lb ae/A (1.37-1.43 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba were <0.02-0.667 (<0.239-<0.504), <0.0711-0.777 (<0.0836-0.707), and <0.0471-0.762 (<0.0594-0.623) ppm, respectively, in/on **corn forage** harvested at a PHI of 43-52 days.

Following three applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 1.22-1.28 lb ae/A (1.37-1.43 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba were <0.02-<0.0234 (<0.02-<0.0228), below the LOQ (<0.02), and <0.02-<0.0206 (<0.02-<0.0203) ppm, respectively, in/on **corn grain** harvested at a PHI of 90-99 days. Corresponding residues in/on **corn stover** were <0.0737-<0.453 (<0.0881-<0.430), <0.0306-0.680 (<0.0318-0.675), and <0.02-0.667 (<0.02-0.616) ppm.

No residue decline trials were conducted.

Table B.7.6.1.2-6. Residue Data from Corn Field Trials with Dicamba.[1]

Location: City, State; Year (Trial ID)	Zone	Corn/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
Jefferson, IA; 2010 (R100231)	5	Pioneer P1480HR	2	Forage	1.24 [1.39]	51	0.0113, (0.00731) [<0.0107]	0.656, 0.330 [0.493]	0.667, <0.340 [<0.504]
				Grain		90	ND, ND [<0.01]	(0.00369), (0.00347) [<0.01]	<0.02, <0.02 [<0.02]
				Stover		90	(0.00976), 0.0105 [<0.0103]	0.0637, 0.0919 [0.0778]	<0.0737, 0.102 [<0.0881]
			3	Forage	1.22 [1.37]	51	(0.00265), ND [<0.01]	0.0611, 0.0860 [0.0736]	<0.0711, <0.0960 [<0.0836]
				Grain		90	ND, ND [<0.01]	(0.00217), (0.00249) [<0.01]	<0.02, <0.02 [<0.02]
				Stover		90	(0.00436), (0.00420) [<0.01]	0.0206, 0.0229 [0.0218]	<0.0306, <0.0329 [<0.0318]
			4	Forage	1.23 [1.37]	51	ND, ND [<0.01]	0.0616, 0.0371 [0.0494]	<0.0716, <0.0471 [<0.0594]
				Grain		90	ND, ND [<0.01]	(0.00226), (0.00244) [<0.01]	<0.02, <0.02 [<0.02]
				Stover		90	(0.00280), (0.00226) [<0.01]	(0.00948), (0.00942) [<0.01]	<0.02, <0.02 [<0.02]

Table B.7.6.1.2-6. Residue Data from Corn Field Trials with Dicamba.[1]

Location: City, State; Year (Trial ID)	Zone	Corn/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
Freeborn, MN; 2010 (R100232)	5	Pioneer 38M60	2	Forage	1.27 [1.42]	51	0.0167, 0.0257 [0.0212]	0.395, 0.558 [0.477]	0.412, 0.584 [0.498]
				Grain		92	ND, ND [<0.01]	(0.00691), (0.00702) [<0.01]	<0.02, <0.02 [<0.02]
				Stover		92	0.0193, 0.0211 [0.0202]	0.194, 0.155 [0.175]	0.213, 0.176 [0.195]
			3	Forage	1.27 [1.42]	51	0.0119, 0.0187 [0.0153]	0.268, 0.457 [0.363]	0.280, 0.476 [0.378]
				Grain		92	ND, ND [<0.01]	(0.00696), (0.00838) [<0.01]	<0.02, <0.02 [<0.02]
				Stover		92	0.0216, (0.00771) [<0.0158]	0.170, 0.0784 [0.124]	0.192, <0.0884 [<0.140]
			4	Forage	1.26 [1.42]	51	0.0198, 0.0199 [0.0199]	0.556, 0.650 [0.603]	0.576, 0.670 [0.623]
				Grain		92	ND, ND [<0.01]	(0.00631), (0.00679) [<0.01]	<0.02, <0.02 [<0.02]
				Stover		92	0.0172, 0.0233 [0.0203]	0.178, 0.177 [0.178]	0.195, 0.200 [0.198]
York, NE; 2010 (R100233)	7	4947RB	2	Forage	1.25 [1.39]	52	(0.00294), (0.00450) [<0.01]	0.220, 0.238 [0.229]	<0.230, <0.248 [<0.239]
				Grain		99	ND, ND [<0.01]	ND, ND [<0.01]	<0.02, <0.02 [<0.02]
				Stover		99	(0.00253), (0.00488) [<0.01]	0.0801, 0.138 [0.109]	<0.0901, <0.148 [<0.119]
			3	Forage	1.24 [1.39]	52	(0.00359), (0.00396) [<0.01]	0.245, 0.285 [0.265]	<0.255, <0.295 [<0.275]
				Grain		99	ND, ND [<0.01]	ND, ND [<0.01]	<0.02, <0.02 [<0.02]
				Stover		99	(0.00280), (0.00388) [<0.01]	0.0778, 0.111 [0.0944]	<0.0878, <0.121 [<0.104]
			4	Forage	1.24 [1.39]	52	(0.00578), (0.00648) [<0.01]	0.351, 0.343 [0.347]	<0.361, <0.353 [<0.357]
				Grain		99	ND, ND [<0.01]	(0.00226), ND [<0.01]	<0.02, <0.02 [<0.02]
				Stover		99	(0.00978), 0.0117 [<0.0109]	0.202, 0.238 [0.220]	<0.212, 0.250 [<0.231]
Polk, NE; 2010 (R100234)	11	4947RB	2	Forage	1.28 [1.43]	43	(0.00836), ND [<0.01]	0.454, (0.00748) [<0.232]	<0.464, <0.02 [<0.242]
				Grain		92	ND, ND [<0.01]	0.0121, 0.0134 [0.0128]	<0.0221, <0.0234 [<0.0228]
				Stover		92	(0.00962),	0.398, 0.440	<0.408, 0.453

Table B.7.6.1.2-6. Residue Data from Corn Field Trials with Dicamba.[1]

Location: City, State; Year (Trial ID)	Zone	Corn/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]		
							Dicamba	5-OH Dicamba[4]	Combined[5]
							0.0126 [<0.0113]	[0.419]	[<0.430]
			3	Forage	1.28 [1.43]	43	0.0132, 0.0139 [0.0136]	0.764, 0.623 [0.694]	0.777, 0.637 [0.707]
				Grain		92	ND, ND [<0.01]	(0.00988), (0.00970) [<0.01]	<0.02, <0.02 [<0.02]
				Stover		92	0.0182, 0.0164 [0.0173]	0.662, 0.654 [0.658]	0.680, 0.670 [0.675]
			4	Forage	1.26 [1.41]	43	0.0134, 0.0113 [0.0124]	0.749, 0.467 [0.608]	0.762, 0.478 [0.620]
				Grain		92	ND, ND [<0.01]	0.0106, (0.00979) [<0.0103]	<0.0206, <0.02 [<0.0203]
				Stover		92	0.0131, 0.0194 [0.0163]	0.552, 0.648 [0.600]	0.565, 0.667 [0.616]

[1] End-use product: Plot 2: 4 lb ae/gal (480 g ae/L) soluble concentrate (SL) formulation of dicamba as the 2-(2-aminoethoxy)ethanol (DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137); Plot 3: 4 lb ae/gal (480 g ae/L) SL formulation of dicamba as the diethylenetriamine (DETA) salt (DETA BAS 183 UYH); and Plot 4: 5 lb ae/gal (600 g ae/L) SL formulation as the N,N-Bis-(3-aminopropyl)methylamine (BPMA) salt (BPMA BAS 183 WBH).
[2] Days after last application.
[3] The LOD was 0.002 ppm, and the LOQ was 0.01 ppm for each analyte. Values between the LOD and LOQ are reported in parenthesis. Residues below the LOQ were not converted to parent equivalents. For samples with nonquantifiable residues, combined residues and per trial averages were calculated by the study reviewer using the LOQ for residues <LOQ. For samples with quantifiable residues, combined residues and per trial averages were calculated by the registrant.
[4] Residues of 5-OH dicamba are expressed in parent equivalents as calculated by the registrant using a molecular weight conversion factor of 0.9325.
[5] Combined residues are the sum of dicamba and 5-OH dicamba.

Table B.7.6.1.2-7. Summary of Residues from Corn Field Trials with Dicamba.											
Crop Matrix	Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
					Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Corn forage	4 lb ae/gal DGA SL	1.24-1.28 [1.39-1.43]	43-52	4	<0.02	0.667	<0.239	<0.504	0.370	0.371	0.150
	4 lb ae/gal DETA SL	1.22-1.28 [1.37-1.43]	43-52	4	<0.0711	0.777	<0.0836	0.707	0.327	0.361	0.261
	5 lb ae/gal BAPMA SL	1.23-1.26 [1.37-1.42]	43-52	4	<0.0471	0.762	<0.0594	0.623	0.489	0.415	0.268
Corn grain	4 lb ae/gal DGA SL	1.24-1.28 [1.39-1.43]	90-99	4	<0.02	<0.0234	<0.02	<0.0228	0.0200	0.0207	0.00138
	4 lb ae/gal DETA SL	1.22-1.28 [1.37-1.43]	90-99	4	<0.02	<0.02	<0.02	<0.02	0.02	0.02	NA
	5 lb ae/gal BAPMA SL	1.23-1.26 [1.37-1.42]	90-99	4	<0.02	<0.0206	<0.02	<0.0203	0.02	0.02	0.00015
Corn stover	4 lb ae/gal DGA SL	1.24-1.28 [1.39-1.43]	90-99	4	<0.0737	0.453	<0.0881	<0.430	0.157	0.208	0.155
	4 lb ae/gal DETA SL	1.22-1.28 [1.37-1.43]	90-99	4	<0.0306	0.680	<0.0318	0.675	0.122	0.238	0.295
	5 lb ae/gal BAPMA SL	1.23-1.26 [1.37-1.42]	90-99	4	<0.02	0.667	<0.02	0.616	0.214	0.266	0.251

[1] n = number of field trials.
[2] Values based on total number of samples.
[3] Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation. For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (0.01 ppm). NA = Not applicable.

III. CONCLUSIONS

The corn field trials are considered scientifically acceptable. The results of the side-by-side trials suggest that combined residues of dicamba and 5-OH dicamba resulting from application of the three SL formulations prepared as different amine salts are comparable in corn matrices. Residues following application of the DETA salt and BAPMA salt are similar, but the highest residues occurred resulted from application of the DETA salt in/on corn forage and stover. Residues resulting from both the DETA and BAPMA salts are higher than the DGA salt. Combined residues resulting from application of the DGA, DETA, and BAPMA salts were at or below the LOQ in/on all samples of corn grain.

Following three applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 1.22-1.28 lb ae/A (1.37-1.43 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba were <0.02-0.667 (<0.239-<0.504), <0.0711-0.777 (<0.0836-0.707), and <0.0471-0.762 (<0.0594-0.623) ppm, respectively, in/on **corn forage** harvested at a PHI of 43-52 days.

Following three applications (single preplant followed by two broadcast foliar applications) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side plots at a total rate of 1.22-1.28 lb ae/A (1.37-1.43 kg ae/ha) combined residues (and per trial averages) of dicamba and 5-OH dicamba were

<0.02-<0.0234 (<0.02-<0.0228), below the LOQ (<0.02), and <0.02-<0.0206 (<0.02-<0.0203) ppm, respectively, in/on **corn grain** harvested at a PHI of 90-99 days. Corresponding residues in/on **corn stover** were <0.0737-<0.453 (<0.0881-<0.430), <0.0306-0.680 (<0.0318-0.675), and <0.02-0.667 (<0.02-0.616) ppm, respectively, in/on **corn stover**.

No residue decline trials were conducted.

An acceptable method was used for residue quantitation, and adequate storage stability data are available to support sample storage durations and conditions for all analytes.

REFERENCES

Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger

B.7.6 Residues Resulting from Supervised Trials (Annex IIA 6.3; Annex IIIA 8.3)

B.7.6.1 Residues in Target Crops

B.7.6.1.1. Soybean (Bridging data)

Document ID:	MRID No. 49379305
Report:	Norris, F. (2013) Magnitude of the Residue of Dicamba in Soybean Matrices, Formulation Bridging Study. BASF Study Number 389561. BASF Reg. Doc. No. 2012/7005501. Unpublished study prepared by BASF Crop Protection. 328 p.
Guidelines:	EPA OCSPP Harmonized Test Guideline 860.1500 Crop Field Trials (August 1996) PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 9 – Crop Field Trials PMRA Regulatory Directive DIR2010-05 – Revisions to the Residue Chemistry Crop Field Trial Requirements OECD Guideline 509 Crop Field Trial (September 2009)
GLP Compliance:	No deviations from regulatory requirements were reported which would have an impact on the validity of the study.
Acceptability:	The study is considered scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document, DP# D429868.
Evaluator:	Peter Savoia, Chemist, Registration Action Branch V/VII/Health Effects Division

Note: This Data Evaluation Record (DER) was originally prepared under contract by Versar, Inc. (6850 Versar Center, Springfield, VA 22151; submitted 12/15/14. The DER has been reviewed by HED and revised to reflect current Office of Pesticide Programs (OPP) policies.

EXECUTIVE SUMMARY

BASF Crop Protection has submitted field trial data for dicamba on soybean. Four field trials were conducted in the United States during the 2010 growing season in the North American Free Trade Agreement (NAFTA) Growing Zone 5 (NE, MN, SD, and IA; 1 trial each). The study was submitted to provide bridging data in support of two new salt formulations of dicamba, the 4 lb ae/gal (480 g ae/L) SL formulation containing diethylenetriamine (DETA) salt (BAS 183 UYH) and the 5 lb ae/gal (600 g ae/L) SL formulation containing N,N-bis-(3-aminopropyl)methylamine (BAPMA) salt (BAS 183 WBH), through side-by-side trials conducted with the 4 lb ae/gal (480 g ae/L) SL formulation containing 2-(2-aminoethoxy)ethanol (a.k.a. diglycolamine; DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137).

Each field site consisted of one untreated plot (Plot 1) and three side-by-side treated plots (Plots 2, 3, and 4) reflecting application of three soluble concentrate (SL) formulations of dicamba each containing a different amine salt: the 4 lb ae/gal (480 g ae/L) DGA SL formulation, the 4 lb ae/gal (480 g ae/L) DETA SL formulation, and the 5 lb ae/gal (600 g ae/L) BAPMA SL

formulation. Each treated plot received one preplant soil application at 0.481-0.523 lb acid equivalent (ae)/A (0.539-0.586 kg ae/ha) followed by one foliar broadcast application at 0.965-1.02 lb ae/A (1.08-1.15 kg ae/ha). There was a 119- to 185-day retreatment interval (RTI). Applications were made using ground equipment, in spray volumes of ~19-23 gal/A (181-211 L/ha) of water. A nonionic surfactant (NIS) was added to spray mixtures for each trial. Samples of soybean forage and hay were harvested at the V6 (BBCH 16) growth stage 66-78 days after the preplant application of dicamba. The registrant indicated that hay was allowed to dry in the field for 2-3 days after harvest. Samples of soybean seed were harvested at a preharvest interval (PHI) of 7 days after the foliar application. Total application rates were 0.481-0.523 lb ae/A (0.539-0.586 kg ae/ha) for soybean forage and hay and 1.45-1.54 lb ae/A (1.63-1.73 kg ae/ha) for seed.

All samples were maintained frozen at the testing facility, during shipping to the laboratory, and were stored frozen until extraction for analysis. The field residue samples were stored frozen a maximum of 957 days (31.5 months) for forage, 962 days (31.6 months) for hay, and 872 days (28.7 months) for seed from harvest to extraction. Samples were analyzed within 1-17 days of extraction; sample extracts were stored under refrigeration (if needed) prior to analysis. Adequate storage stability data are available reflecting the stability of residues of dicamba and DCSA during frozen storage for up to 4 months in/on soybean forage and residues of dicamba and 5-OH dicamba are stable during frozen storage for up to 3 and 2 years, respectively, in/on corn forage, silage, grain, and fodder (refer to the Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger). Although the available storage stability data for DCSA do not support the storage duration of the sample in this study, given the similar chemical structure of DCSA to the parent compound and the DCSA residues found in this study it may be concluded that potential DCSA residues were unaffected by frozen storage prior to sample analysis. Data for dicamba and 5-OH dicamba are acceptable to support the storage conditions and durations of samples from the submitted field trials.

Samples were analyzed for residues of dicamba and its metabolites, dichlorosalicyclic acid (DCSA) and 5-OH dicamba, using high pressure liquid chromatography with tandem mass spectrometric detection (LC/MS/MS); Method D0902 with minor modifications. The limit of quantitation (LOQ; determined as the lowest limit of method validation, LLMV) was 0.01 ppm for all analytes in the soybean seed matrices. The LOQ was 0.05 ppm for all analytes in the soybean forage and hay matrices. Acceptable concurrent recoveries were reported for samples fortified with dicamba, 5-OH dicamba, and DCSA at 0.01-10 ppm. The fortification levels used in concurrent method recovery were adequate to bracket expected residue levels. Concurrent recoveries were not corrected for apparent residues in controls; residues in the treated samples were not corrected for residues in controls. Quantifiable residues of DCSA and 5-OH dicamba were converted to parent equivalents by the registrant using molecular weight conversion factor of 1.068 and 0.9325, respectively.

When the trial results are compared on a site-by-site basis, it appears that the residues resulting from application of the DGA salt and the DETA salt formulations are comparable in/on soybean seed, and that the highest residues occurred following application of the DETA salt. Residues resulting from both the DGA and DETA salts are higher than the BAPMA salt. Combined residues of dicamba, 5-OH dicamba, and DCSA resulting from application of the DGA, DETA, and BAPMA salts were below the LOQ in/on all samples of soybean forage and hay.

Following a single preplant application of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side trials at a rate of 0.481-0.523 lb ae/A (0.539-0.586 kg ae/ha), combined residues (and per trial averages) of dicamba, 5-OH dicamba, and DCSA were below the combined LOQ (<0.15 ppm) in/on **soybean forage** and **hay** harvested at a PHI of 66-78 days (V6; BBCH 16 growth stage). The absence of residues demonstrates that measurable residues are not drawn into the soybean foliage following a preplant soil application.

Following two applications (single preplant application followed by one broadcast foliar application) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side trials at a total rate of 1.45-1.54 lb ae/A (1.63-1.73 kg ae/ha) combined residues (and per trial averages) of dicamba, 5-OH dicamba, and DCSA were <0.03-6.27 (<0.03-5.82), <0.03-7.00 (<0.03-6.84), and <0.03-2.77 (<0.03-<1.42) ppm, respectively, in/on **soybean seed** harvested at a PHI of 7 days. The registrant noted that several soybean seed samples had significant residues but not in a particular pattern and provided a couple of explanations for these residues. First, it was suggested that the pod containing the seed may have begun to split allowing residues to contact the seed. Another possibility is contact of the seed with the outer surface of the pods during shelling.

No residue decline trials were conducted.

I. MATERIALS AND METHODS

A. MATERIALS

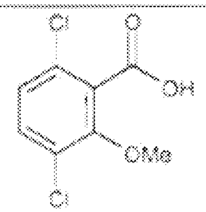
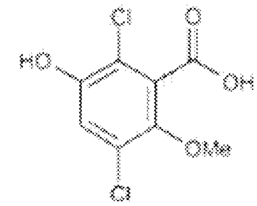
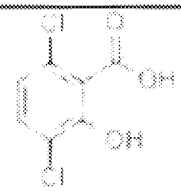
Table B.7.6.1.1-1. Nomenclature for Dicamba.	
Common name	Dicamba
Identity	3,6-dichloro-2-methoxybenzoic acid
CAS no.	1918-00-9 (dicamba acid) or 1982-69-0 (sodium salt of dicamba)
Company experimental name	BAS 183 H
Other synonyms (if applicable)	N/A
	
Common name	5-Hydroxy-dicamba
Identity	3,6-dichloro-5-hydroxy-2-methoxy-benzoic acid
CAS no.	7600-50-2
Company experimental name	5-OH dicamba
Other synonyms (if applicable)	N/A

Table B.7.6.1.1-1. Nomenclature for Dicamba.



Common name	3,6-dichlorosalicylic acid
Identity	3,6-dichloro-2-hydroxybenzoic acid
CAS no.	3401-80-7
Company experimental name	DCSA
Other synonyms (if applicable)	N/A



B. Study Design

1. Test Procedure

A total of four side-by-side residue trials in/on soybean were conducted with three 4 or 5 lb ae/gal SL formulations during the 2010 growing season. Field trial locations by NAFTA growing zone are summarized in Table B.7.6.1.1.2.

Table B.7.6.1.1-2. Trial Numbers and Geographical Locations.

Crop	No. Trials	NAFTA Growing Zone												Total
		1	2	3	4	5	6	7	8	9	10	11	12	
Soybean	Sub.					4								4
	Req.[1]		2/2		3/2	15/11								20/15

[1] As per Table 5 of 860.1500 for soybeans; the second number reflects a 25% reduction in the number of field trials allowed for the crop as a representative commodity in support of a crop group/subgroup tolerance or when application results in no quantifiable residues.

Locations and detailed use patterns for the trials are provided in Table B.7.6.1.1-3.

Table B.7.6.1.1-3. Study Use Pattern.

Location: City, State; Year (Trial ID)	Plot	End-use Product[1]	Method of Application/ Timing of Application	Volume (gal/A) [L/ha]	Rate per Application (lb ae/A) [kg ae/ha]	Retreatment Interval (days)	Total Rate[2] (lb ae/A) [kg ae/ha]	Surfactant/ Adjuvant[3]
York, NE; 2010 (R100188)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	20.36 [190.4]	0.4992 [0.5595]	--	0.4992 [0.5595]	NIS
			2. Broadcast foliar spray; BBCH 89	20.18 [188.7]	1.0235 [1.1471]	148	1.5227 [1.7066]	NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	20.39 [190.6]	0.5000 [0.5604]	--	0.5000 [0.5604]	NIS
			2. Broadcast foliar spray; BBCH 89	19.81 [185.3]	1.0048 [1.1261]	148	1.5048 [1.6865]	NIS

Table B.7.6.1.1-3. Study Use Pattern.								
Location: City, State; Year (Trial ID)	Plot	End-use Product[1]	Method of Application/ Timing of Application	Volume (gal/A) [L/ha]	Rate per Application (lb ae/A) [kg ae/ha]	Retreatment Interval (days)	Total Rate[2] (lb ae/A) [kg ae/ha]	Surfactant/ Adjuvant[3]
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	20.33 [190.1]	0.4995 [0.5598]	--	0.4995 [0.5598]	NIS
			2. Broadcast foliar spray; BBCH 89	19.71 [184.3]	0.9998 [1.1208]	148	1.4993 [1.6806]	NIS
Sterns, MN; 2010 (R100189)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	20.03 [187.3]	0.4807 [0.5387]	--	0.4807 [0.5387]	NIS
			2. Broadcast foliar spray; BBCH 89	20.21 [189.0]	1.0105 [1.1325]	185	1.4912 [1.6712]	NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	20.21 [189.0]	0.4850 [0.5435]	--	0.4850 [0.5435]	NIS
			2. Broadcast foliar spray; BBCH 89	20.03 [187.3]	1.0015 [1.1224]	185	1.4865 [1.6659]	NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	20.30 [189.9]	0.5075 [0.5688]	--	0.5075 [0.5688]	NIS
			2. Broadcast foliar spray; BBCH 89	20.12 [188.2]	1.0060 [1.1275]	185	1.5135 [1.6963]	NIS
Turner, SD; 2010 (R100190)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	20.14 [188.36]	0.4995 [0.5598]	--	0.4995 [0.5598]	NIS
			2. Broadcast foliar spray; BBCH 96	20.04 [187.43]	0.9993 [1.1200]	145	1.4988 [1.6798]	NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	19.93 [186.39]	0.4943 [0.5540]	--	0.4943 [0.5540]	NIS
			2. Broadcast foliar spray; BBCH 96	20.16 [188.55]	1.0053 [1.1267]	145	1.4996 [1.6807]	NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	19.65 [183.78]	0.4873 [0.5461]	--	0.4873 [0.5461]	NIS
			2. Broadcast foliar spray; BBCH 96	19.40 [181.44]	0.9668 [1.0835]	145	1.4541 [1.6296]	NIS
Guthrie, IA; 2010 (R100191)	2	4 lb ae/gal DGA SL	1. Soil broadcast spray; preplant	20.22 [189.11]	0.4860 [0.5447]	--	0.4860 [0.5447]	NIS
			2. Broadcast foliar spray; BBCH 95	20.69 [193.51]	0.9647 [1.0812]	119	1.4507 [1.6259]	NIS
	3	4 lb ae/gal DETA SL	1. Soil broadcast spray; preplant	20.33 [190.14]	0.4887 [0.5477]	--	0.4887 [0.5477]	NIS
			2. Broadcast foliar spray; BBCH 95	21.40 [200.15]	0.9696 [1.0867]	119	1.4583 [1.6344]	NIS
	4	5 lb ae/gal BAPMA SL	1. Soil broadcast spray; preplant	20.87 [195.19]	0.5231 [0.5862]	--	0.5231 [0.5862]	NIS
			2. Broadcast foliar spray; BBCH 95	22.60 [211.37]	1.0172 [1.1400]	119	1.5403 [1.7262]	NIS

[1] EP = End-use product; Plot 2: 4 lb ae/gal (480 g ae/L) soluble concentrate (SL) formulation of dicamba as the 2-(2-aminoethoxy)ethanol (DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137); Plot 3: 4 lb ae/gal (480 g ae/L) SL formulation of dicamba as the diethylenetriamine (DETA) salt (DETA BAS 183 UYH); and Plot 4: 5 lb ae/gal (600 g ae/L) SL formulation as the N,N-Bis-(3-aminopropyl)methylamine (BAPMA) salt (BAS 183 WBH).
[2] Forage and hay were harvested after a single application.
[3] NIS = nonionic surfactant.

Soybean was grown and maintained according to typical agricultural practices. Irrigation was used at the NE and MN trial sites. No unusual weather conditions were reported to have adversely affected crop production or yield during the study; however, forage and hay samples from the MN trial were lost in a fire.

Sample Handling and Preparation

Single control and duplicate treated samples of soybean forage and hay were harvested at a PHI of 66-78 days after the preplant application at growth stage targeting V6 (BBCH 16). The registrant indicated that hay samples were allowed to dry in the field 2-3 days prior to collection. Single control and duplicate treated samples of soybean seed were harvested at maturity 7 days after the second application. The samples were commercially acceptable soybean RAC samples and consisted of at least 12 plants. Each RAC sample weighed ≥1 kg (forage, hay or seed). All treated samples were placed in frozen storage at the field trials immediately after collection. All samples were shipped within 62 days of collection by ACDS freezer truck to BASF Agricultural Research Center (Research Triangle Park, NC) for homogenization, where they were maintained frozen (<-5 °C) prior to extraction for analysis.

2. Description of Analytical Procedures

Samples of soybean (forage, hay, and seed) were analyzed for residues of dicamba, DCSA, and 5-OH dicamba using a modified version of LC/MS/MS method D0902. For analysis of soybean forage and hay, minor modifications to instrumentation and conditions were made including: (i) after the extract is brought to a final volume of 100 mL, a hexane partition is added before the hexane/ethyl acetate partition. This additional clean-up of the samples was necessary due to matrix interferences in forage and hay; (ii) the reference standard solutions and the sample dilution solution were prepared in 10:90 methanol:water plus 0.1% formic acid to improve the LC/MS/MS response; and (iii) two soybean seed analytical sets were run with these modified LC/MS/MS conditions to address matrix interferences observed in forage and hay chromatography. A brief description of the method was included in the submission; for a complete description and method validation refer to 48001303.DER, A. Kamel, 4/30/13.

Briefly, homogenized samples were heated with 1 N HCl at ~90 °C for ~45 minutes. The extract was cooled to room temperature and filtered, then adjusted to volume with water. The extract was adjusted to pH 9-10 with concentrated NH_4OH, vortexed, and adjusted to pH 3-4 with concentrated formic acid. Sodium chloride was added, and the extract was partitioned twice with hexane:ethyl acetate (1:1, v:v) followed by centrifugation. The resulting organic phase was reduced to dryness under nitrogen, then reconstituted in methanol:water (10:90, v:v) for analysis by LC/MS/MS. Two transition ions were monitored for each analyte.

The LOQ (determined as the LLMV), was 0.01 ppm for all analytes in the soybean seed; the corresponding limit of detection (LOD) was 0.002 ppm. The LOQ was 0.05 ppm for all analytes in the soybean forage and hay; the corresponding LOD was 0.01 ppm.

II. RESULTS AND DISCUSSION

Method performance was evaluated by use of concurrent recovery samples. Samples of soybean forage were fortified with each analyte at 0.01, 0.05, and 0.1 ppm. Samples of soybean hay were fortified with each analyte at 0.01, 0.05, and 1.0 ppm. Samples of soybean seed were fortified with each analyte at 0.01, 0.05 (5-OH dicamba only), 1.0, and 10 ppm. Average recoveries were within the acceptable range of 70-120% with the exception of soybean forage and hay at the LLMV (0.01 ppm). Based on the level of interference observed in the chromatography for forage and hay, and some low recoveries at the LLMV particularly for 5-OH dicamba and DCSA in hay, the LOQ for forage and hay was considered to be 0.05 ppm because that was the level for which acceptable recoveries were obtained consistently for each analyte. The method was considered valid for the analysis of dicamba residues in/on soybean matrices (Table B.7.6.1.1-4). The fortification levels bracketed the measured residues. Concurrent recoveries were not corrected for apparent residues in controls; residues in the treated samples were not corrected for apparent residues in controls.

For analysis of soybean forage, hay, and seed, the detector response was linear (coefficient of determination, $r^2 \geq 0.9983$ for dicamba, $r^2 \geq 0.9918$ for DCSA, and $r^2 \geq 0.9898$ for 5-OH dicamba within the range of 0.1-2.50 ng/mL). Representative chromatograms of control samples, fortified samples, and treated samples were provided. The control chromatograms generally had no peaks of interest above the chromatographic background. The fortified sample chromatograms contained only the analyte of interest, and peaks were symmetrical and well defined. Apparent residues of each analyte in controls were LOQ (<0.01 ppm for seed and <0.05 ppm for forage and hay). The reported residue values were not corrected for apparent residues in controls. Residues of DCSA and 5-OH dicamba are expressed in parent equivalents as calculated by the registrant using a molecular weight conversion factor of 1.068 and 0.9325, respectively.

Table B.7.6.1.1-4. Summary of Procedural/Concurrent Recoveries of Dicamba, 5-OH Dicamba, and DCSA from Soybean Matrices.

Matrix	Analyte	Fortification Level (ppm)	Sample size (n)	Recoveries[1] (%)	Mean ± Std. Dev.[2] (%)
Concurrent Recoveries					
Soybean forage	Dicamba	0.01	4	61, 67; 70, 71	67 ± 4
		0.05	2	88, 98	93
		0.1	1	92	92
	5-OH Dicamba	0.01	6	68; 72-94	82 ± 10
		0.05	2	102, 82	92
		0.1	2	90, 92	91
	DCSA	0.01	6	67; 76-87	80 ± 8
		0.05	2	69; 105	87
		0.1	2	83, 91	87

Table B.7.6.1.1-4. Summary of Procedural/Concurrent Recoveries of Dicamba, 5-OH Dicamba, and DCSA from Soybean Matrices.

Matrix	Analyte	Fortification Level (ppm)	Sample size (n)	Recoveries[1] (%)	Mean ± Std. Dev.[2] (%)
Soybean hay	Dicamba	0.01	2	72, 100	86
		0.05	2	81, 81	81
		1.0	1	73	73
	5-OH Dicamba	0.01	4	53, 62, 65; 86	66 ± 14
		0.05	4	75-87	82 ± 6
		1.0	1	93	93
	DCSA	0.01	3	55, 57; 71	61 ± 9
		0.05	3	67; 77, 76	73 ± 5
		1.0	1	77	77
Soybean seed	Dicamba	0.01	6	78-110	91 ± 12
		1.0	5	74[3], 71, 87[3], 81[3], 72	77 ± 7
		10	1	83[3]	83
	5-OH Dicamba	0.01	4	63, 65; 81, 82	73 ± 10
		0.05	2	102, 99[3]	100
		1.0	1	85[3]	85
		10	1	94[3]	94
	DCSA	0.01	6	71-102	87 ± 11
		1.0	5	78-83[4]	80 ± 2
		10	1	88[3]	88

[1] Concurrent recoveries were not corrected for apparent residues in controls.
[2] Standard deviation is not calculated for sample sizes <3.
[3] Mean value of duplicated injections of the same fortification sample.
[4] Includes mean value of duplicated injections of the same fortification sample.

The field residue samples were stored frozen a maximum of 957 days (31.5 months) for forage, 962 days (31.6 months) for hay, and 872 days (28.7 months) for seed from harvest to extraction (Table B.7.6.1.1-5). Samples were analyzed within 1-17 days of extraction; sample extracts were stored under refrigeration (if needed) prior to analysis. Adequate storage stability data are available reflecting the stability of residues of dicamba and DCSA during frozen storage for up to 4 months in/on soybean forage and residues of dicamba and 5-OH dicamba are stable during frozen storage for up to 3 and 2 years, respectively, in/on corn forage, silage, grain, and fodder (refer to the Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger). Although the available storage stability data for DCSA do not support the storage duration of the sample in this study, given the similar chemical structure of DCSA to the parent compound and the DCSA residues found in this study, it may be concluded that potential DCSA residues were unaffected by frozen storage prior to sample analysis. Data for dicamba and 5-OH dicamba are acceptable to support the storage conditions and durations of samples from the submitted field trials.

Table B.7.6.1.1-5. Summary of Storage Conditions.			
Matrix	Storage Temperature (°C)	Actual Storage Duration[1]	Interval of Demonstrated Storage Stability
Soybean forage	≤-5	885-957 days (29.1-31.5 months)	Adequate storage stability data are available reflecting the stability of residues of dicamba and DCSA during frozen storage for up to 4 months in/on soybean forage. In addition, dicamba and 5-OH dicamba are stable during frozen storage for up to 3 and 2 years, respectively, in/on corn forage, silage, grain, and fodder.[2]
Soybean hay		878-962 days (28.9-31.6 months)	
Soybean seed		641-872 days (21.1-28.7 months)	

[1] Interval from harvest to extraction. Samples were analyzed within 1-17 days of extraction.
[2] Refer to the Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger.

The results from the submitted field trials are presented in Tables B.7.6.1.1-6 and B.7.6.1.1-7. When the trial results are compared on a site-by-site basis, it appears that the residues resulting from application of the DGA salt and the DETA salt formulations are comparable in/on soybean seed, and that the highest residues occurred following application of the DETA salt. Residues resulting from both the DGA and DETA salts are higher than the BAPMA salt. Combined residues of dicamba, 5-OH dicamba, and DCSA resulting from application of the DGA, DETA, and BAPMA salts were below the LOQ in/on all samples of soybean forage and hay.

Following a single preplant application of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side trials at a rate of 0.481-0.523 lb ae/A (0.539-0.586 kg ae/ha), combined residues (and per trial averages) of dicamba, 5-OH dicamba, and DCSA were below the combined LOQ (<0.15 ppm) in/on **soybean forage** and **hay** harvested at a PHI of 66-78 days (V6; BBCH 16 growth stage). The absence of residues demonstrates that measurable residues are not drawn into the soybean foliage following a preplant soil application.

Following two applications (single preplant application followed by one broadcast foliar application) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side trials at a total rate of 1.45-1.54 lb ae/A (1.63-1.73 kg ae/ha) combined residues (and per trial averages) of dicamba, 5-OH dicamba, and DCSA were <0.03-6.27 (<0.03-5.82), <0.03-7.00 (<0.03-6.84), and <0.03-2.77 (<0.03-<1.42) ppm, respectively, in/on **soybean seed** harvested at a PHI of 7 days. The registrant noted that several soybean seed samples had significant residues but not in a particular pattern and provided a couple of explanations for these residues. First, it was suggested that the pod containing the seed may have begun to split allowing residues to contact the seed. Another possibility is contact of the seed with the outer surface of the pods during shelling.

No residue decline trials were conducted.

Table B.7.6.1.1-6. Residue Data from Soybean Field Trials with Dicamba.[1]										
Location: City, State; Year (Trial ID)	Zone	Soybean/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]			
							Dicamba	DCSA[4]	5-OH Dicamba[4]	Combined[5]
York, NE; 2010 (R100188)	5	93Y12	2	Forage	0.4992 [0.5595]	66	ND, ND [<0.05]	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Hay		74	<0.05[6], ND[6] [<0.05]	ND, ND [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Seed	1.5227 [1.7066]	7	0.103, 0.107 [0.105]	ND, ND [<0.01]	ND, ND [<0.01]	<0.123, <0.127 [<0.125]
			3	Forage	0.5000 [0.5604]	66	ND, ND [<0.05]	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Hay		74	<0.05[6], ND[6] [<0.05]	ND, ND [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Seed	1.5048 [1.6865]	7	0.0940, 0.111 [0.103]	ND, ND [<0.01]	ND, ND [<0.01]	<0.114, <0.131 [0.123]
			4	Forage	0.4995 [0.5598]	66	ND, ND [<0.05]	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Hay		74	ND[6], ND[6] [<0.05]	ND, ND [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Seed	1.4993 [1.6712]	7	0.107, 0.134 [0.121]	ND, ND [<0.01]	ND, ND [<0.01]	<0.127, <0.154 [<0.141]
Sterns, MN; 2010 (R100189)	5	Asgrow AG1002	2	Forage	0.4807 [0.5387]	Samples lost in fire				
				Hay		Samples lost in fire				
				Seed	1.4912 [1.6712]	7	ND, ND [<0.01]	ND, ND [<0.01]	ND, ND [<0.01]	<0.03, <0.03 [<0.03]
			3	Forage	0.4850 [0.5435]	Samples lost in fire				
				Hay		Samples lost in fire				
				Seed	1.4865 [1.6659]	7	ND, ND [<0.01]	ND, ND [<0.01]	ND, ND [<0.01]	<0.03, <0.03 [<0.03]
			4	Forage	0.5075 [0.5688]	Samples lost in fire				
				Hay		Samples lost in fire				
				Seed	1.5135 [1.6963]	7	ND, ND [<0.01]	ND, ND [<0.01]	ND, ND [<0.01]	<0.03, <0.03 [<0.03]

Table B.7.6.1.1-6. Residue Data from Soybean Field Trials with Dicamba.[1]

Location: City, State; Year (Trial ID)	Zone	Soybean/ Variety	Plot	Matrix	Rate (lb ae/A) [kg ae/ha]	PHI [2] (days)	Residues[3] (ppm) [Average]			
							Dicamba	DCSA[3]	5-OH Dicamba[4]	Combined[5]
Turner, SD; 2010 (R100190)	5	Hefty 6238359	2	Forage	0.4995 [0.5598]	67	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Hay		74	ND, ND [<0.05]	ND, ND [<0.05]	ND, ND [<0.05]	<0.15, <0.15 [<0.15]
				Seed	1.4988 [1.6963]	7	5.01, 5.84 [5.43]	0.195, 0.233 [0.214]	0.159, 0.199 [0.179]	5.36, 6.27 [5.82]
			3	Forage	0.4943 [0.5540]	67	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Hay		74	ND, ND [<0.05]	ND, ND [<0.05]	ND, ND [<0.05]	<0.15, <0.15 [<0.15]
				Seed	1.4996 [1.6807]	7	0.0781, 0.0203 [0.0492]	ND, ND [<0.01]	ND, 0.0533 [<0.0313]	<0.0981, <0.0836, [<0.0909]
			4	Forage	0.4873 [0.5461]	67	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Hay		74	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	ND[6], ND[6] [<0.05]	<0.15[6], <0.15[6] [<0.15]
				Seed	1.4541 [1.6296]	7	0.0227, 0.0252 [0.0240]	ND, ND [<0.01]	ND, ND [<0.01]	<0.0427, <0.0452 [<0.0440]
Guthrie, IA; 2010 (R100191)	5	93Y13	2	Forage	0.4860 [0.5447]	78	ND, ND [<0.05]	ND, ND [<0.05]	ND, ND [<0.05]	<0.15, <0.15 [<0.15]
				Hay		78	ND, ND [<0.05]	ND, ND [<0.05]	ND, ND [<0.05]	<0.15, <0.15 [<0.15]
				Seed	1.4507 [1.6259]	7	0.0401[6], 0.0448[6] [0.0424]	ND[6], ND[6] [<0.01]	ND, ND [<0.01]	<0.0601[6], <0.0648[6] [<0.0625]
			3	Forage	0.4887 [0.5477]	78	ND, ND [<0.05]	ND, ND [<0.05]	ND, ND [<0.05]	<0.15, <0.15 [<0.15]
				Hay		78	ND, ND [<0.05]	ND, ND [<0.05]	ND, ND [<0.05]	<0.15, <0.15 [<0.15]
				Seed	1.4583 [1.6344]	7	5.75[6], 5.50[6] [5.63]	0.437[6], 0.402[6] [0.420]	0.812[6], 0.775[6] [0.794]	7.00[6], 6.68[6], [6.84]
			4	Forage	0.5231 [0.5862]	78	ND, ND [<0.05]	ND, ND [<0.05]	ND, ND [<0.05]	<0.15, <0.15 [<0.15]
				Hay		78	ND, ND [<0.05]	ND, ND [<0.05]	ND, ND [<0.05]	<0.15, <0.15 [<0.15]
				Seed	1.5403 [1.7262]	7	0.0298[6], 2.63[6] [1.33]	ND[6], 0.103[6] [<0.0566]	<0.0371[6], 0.0340[6] [<0.0355]	<0.0769[6], 2.77[6] [<1.42]

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[1] End-use product: Plot 2: 4 lb ae/gal (480 g ae/L) soluble concentrate (SL) formulation of dicamba as the 2-(2-aminoethoxy)ethanol (DGA) salt (BAS 183-09; Clarity Herbicide; EPA Reg. No. 7969-137); Plot 3: 4 lb ae/gal (480 g ae/L) SL formulation of dicamba as the diethylenetriamine (DETA) salt (DETA BAS 183 UYH); and Plot 4: 5 lb ae/gal (600 g ae/L) SL formulation as the N,N-Bis-(3-aminopropyl)methylamine (BPMA) salt (BAS 183 WBH).
[2] For forage and hay, days after preplant application. For seed, days after foliar application.
[3] ND = Not detected (<LOD). The LOD was 0.01 ppm (forage and hay) and 0.002 ppm (seed), and the LOQ was 0.05 ppm (forage and hay) and 0.01 ppm (seed) for each analyte. Residues below the LOQ were not converted to parent equivalents. For samples with nonquantifiable residues, combined residues and per trial averages were calculated by the study reviewer using the LOQ for residues <LOQ. For samples with quantifiable residues, combined residues and per trial averages were calculated by the registrant.
[4] Residues of DCSA and 5-OH dicamba are expressed in parent equivalents as calculated by the registrant using a molecular weight conversion factor of 1.068 and 0.9325, respectively.
[5] Combined residues are the sum of dicamba, DCSA, and 5-OH dicamba.
[6] Mean value of duplicate injections of a single sample.

Table B.7.6.1.1-7. Summary of Residues from Soybean Field Trials with Dicamba.

Crop Matrix	Formulation	Total Application Rate (lb ae/A) [kg ae/ha]	PHI (days)	n[1]	Combined Residues (ppm parent equivalents)						
					Min.[2]	Max.[2]	LAFT[3]	HAFT[3]	Median[3]	Mean[3]	SD[3]
Soybean forage	4 lb ae/gal DGA SL	0.4807-0.4995 [0.5387-0.5598]	66-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
	4 lb ae/gal DETA SL	0.4850-0.5000 [0.5435-0.5604]	66-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
	5 lb ae/gal BAPMA SL	0.4873-0.5231 [0.5461-0.5862]	66-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
Soybean hay	4 lb ae/gal DGA SL	0.4807-0.4995 [0.5387-0.5598]	74-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
	4 lb ae/gal DETA SL	0.4850-0.5000 [0.5435-0.5604]	74-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
	5 lb ae/gal BAPMA SL	0.4873-0.5231 [0.5461-0.5862]	74-78	3	<0.15	<0.15	<0.15	<0.15	0.15	0.15	NA
Soybean seed	4 lb ae/gal DGA SL	1.4507-1.5227 [1.6296-1.7066]	7	4	<0.03	6.27	<0.03	5.82	0.0937	1.51	2.87
	4 lb ae/gal DETA SL	1.4583-1.5048 [1.6344-1.6865]	7	4	<0.03	7.00	<0.03	6.84	0.107	1.770	3.38
	5 lb ae/gal BAPMA SL	1.4541-1.5403 [1.6296-1.7262]	7	4	<0.03	2.77	<0.03	1.42	0.0922	0.409	0.677

[1] n = number of field trials.
[2] Values based on total number of samples.
[3] Values based on per-trial averages. LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation. For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (0.05 ppm for forage and hay and 0.01 ppm for seed). NA = Not applicable.

III. CONCLUSIONS

The soybean field trials are considered scientifically acceptable. When the trial results are compared on a site-by-site basis, it appears that the residues resulting from application of the DGA salt and the DETA salt formulations are comparable in/on soybean seed, and that the highest residues occurred following application of the DETA salt. Residues resulting from both the DGA and DETA salts are higher than the BAPMA salt. Combined residues of dicamba, 5-OH dicamba, and DCSA resulting from application of the DGA, DETA, and BAPMA salts were below the LOQ in/on all samples of soybean forage and hay.

Following a single preplant application of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side trials at a rate of 0.481-0.523 lb ae/A (0.539-0.586 kg ae/ha), combined residues (and per trial averages) of dicamba, 5-OH dicamba, and DCSA were below the combined LOQ (<0.15 ppm) in/on **soybean forage** and **hay** harvested at a PHI of 66-78 days (V6; BBCH 16 growth stage). The absence of residues demonstrates that measurable residues are not drawn into the soybean foliage following a preplant soil application.

Following two applications (single preplant application followed by one broadcast foliar application) of the 4 lb ae/gal (480 g ae/L) DGA SL, 4 lb ae/gal (480 g ae/L) DETA SL, and 5 lb ae/gal (600 g ae/L) BAPMA SL formulations in side-by-side trials at a total rate of 1.45-1.54 lb ae/A (1.63-1.73 kg ae/ha) combined residues (and per trial averages) of dicamba, 5-OH dicamba, and DCSA were <0.03-6.27 (<0.03-5.82), <0.03-7.00 (<0.03-6.84), and <0.03-2.77 (<0.03-<1.42) ppm, respectively, in/on **soybean seed** harvested at a PHI of 7 days. The registrant noted that several soybean seed samples had significant residues but not in a particular pattern and provided a couple of explanations for these residues. First, it was suggested that the pod containing the seed may have begun to split allowing residues to contact the seed. Another possibility is contact of the seed with the outer surface of the pods during shelling.

No residue decline trials were conducted.

An acceptable method was used for residue quantitation, and adequate storage stability data are available to support sample storage durations and conditions for all analytes.

REFERENCES

48001303.DER, A. Kamel, 4/30/13
Residue Chemistry Chapter of the Dicamba RED, DP# 317699, 12/20/05, C. Olinger